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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at 26 Portland Place, London, W.1, on 5 December 1956, the Chair being taken by Dr E. B. Evans, a vice-president of the Institute.

The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting.

The Chairman, introducing the author of the paper to be presented, said: Mr Cooke is a physical chemist by training, and the fact that he is so indicates the close connexion between some aspects of physical chemistry and the problems of the field engineer. He joined the British Petroleum Company some fourteen or fifteen years ago, and has been engaged in a variety of problems connected with physical chemistry and its applications, including X-ray crystallography and the examination of clays and catalysts.

The following paper was then presented in summary.

THE HYDRAULICS OF DRILLING MUD FLOW*

with particular reference to annular flow and caving hole difficulties

By P. W. COOKE† (Associate Fellow)

SUMMARY

The problem of removing caved debris from a well bore is discussed and calculations are given to demonstrate that turbulent flow in the annulus is desirable. The removal of drilled cuttings presents no problem under either turbulent or laminar flow conditions for any mud weight as long as a minimum annular space velocity of about 1 ft/sec is maintained. If laminar flow has to be accepted on account of pump pressure limitations, it would seem desirable to keep the yield value and plastic viscosity of the mud to a minimum and increase the mud weight within reasonable limits.

INTRODUCTION

THE question of mud flow in a well annulus has received considerable attention in recent years, and several papers in the literature have discussed the problems of the carrying capacity of mud and the optimum flow type required for the most efficient removal of drilled debris.[1–5] Laboratory and field experiments designed to simulate the transport of cuttings in an ascending column of mud have been described, but generally the actual rheological properties of the muds used have not been quoted.

Many of the problems associated with specific mud properties have disappeared with the advent of high quality muds possessing low fluid loss characteristics and an inhibited filtrate attack on the formation penetrated. One important problem remains, however, which is closely related to the rheological properties of a mud. This is caving hole difficulties, which are very frequently encountered when a well penetrates thick shale sections. The nature of this problem was discussed in a paper [6] at the Fourth World Petroleum Congress, although no experimental data were included. In general, previous papers have been concerned only with the transport of drilled debris, the particle size of which is small and the removal of which presents few problems.

When caving conditions are encountered the main problem is the removal of debris considerably larger than drilled cuttings. Caved fragments have been observed in the field to be as large as 4 inches average diameter and 1 to 2 inches thick. Three factors influence the amount of caving that takes place and they are: (1) the geological structure of the formation; (2) the chemical and physical properties of the mud fluid; and (3) the drilling technique. Caving can be frequently stopped by attention to the mud fluid and drilling technique, although satisfactory improvement may not be obtained in the presence of abnormal formation pressures, faulting, and steeply dipping beds.

If, in spite of remedial action, caving still persists, the caved debris must be removed quickly in order to eliminate the possibility of stuck tools. In this connexion it cannot be too strongly emphasized that open-hole time exerts a great influence on this type of caving, a rapid deterioration in hole conditions frequently accompanying prolonged open-hole time. The method of removing the caved debris as quickly as possible is the subject of much speculation and is one which presents the field mud engineer with a problem, the solution of which appears to rest on two contradictory beliefs.

From hydraulic considerations, an increase in annulus velocity must contribute to more efficient removal of debris, and in this respect turbulent flow is to be preferred, since, under such flow conditions, the caved fragments present their surfaces of maximum area to the direction of flow, of which the velocity distribution is uniform at all points in the annulus. According to some authorities, however, the flow eddies associated with turbulent flow result in further disruption of the borehole wall and thus aggravate the problem. A preference for laminar

* MS received 23 January 1956.

† The British Petroleum Co. Ltd.

type flow is thus indicated, the velocity distribution of which varies from a maximum at some point near the centre of the annulus ring to zero at the walls of the hole, and near the drill pipe. Dead areas of flow are then created, especially in enlarged portions of the hole, into which the caved debris is carried hydraulically and deposited. When flow ceases the deposited particles then tend to fall into the hole and stick the tools.

When caving difficulties persist, the main problems are to decide which type of mud flow is preferable; how a change-over from one flow type to another can be achieved without aggravating the hole condition; and, finally, what side effects are likely to be introduced by the change-over. This paper attempts to show that: (1) turbulent flow is to be preferred for the most efficient removal of caved debris; (2) it is seldom achieved in the field with the usual rates of circulation employing the more conventional mud types; and (3) the currently accepted practice of increasing the viscosity of the mud to aid in removing caved debris only serves to aggravate its accumulation.

It must be reiterated that these statements are meant to apply only in the case of caving brought about by the existence of abnormal conditions mentioned previously and are not meant to apply to caving induced by incorrect mud type application or bad drilling practice.

That increasing the mud viscosity, according to reported statements in the literature, has contributed to better hole conditions is not denied, but it is suggested that the improvement is due to side effects introduced when the various methods of increasing viscosity are carried out. For example, two of the commoner methods of increasing viscosity are the introduction of oil into the mud or addition of quantities of starches or carboxy-methylcellulose. In both cases a reduction in fluid loss of the mud usually results, and this reduction may in itself be responsible for the improvement, by reduced hydration of the shale.

EXPERIMENTAL WORK

The rheological properties, yield point and plastic viscosity, of a series of lime muds varying in weight from 1·2 to 2·5 sp. gr. were determined employing a funnel type viscometer described in the paper by Beck, Nuss, and Dunn.[2] The weighted muds were prepared by the direct addition of barytes to a base mud compounded as follows:

Water	1000 ml
Bentonite	80 g
Quebracho	5 g
Caustic soda	8 g
Lime	16 g

Table I summarizes the test results. These figures for laboratory muds were found to be reproducible in the field, and Table II shows the corresponding properties for three field muds taken from circulation and tested in the laboratory.

Realizing that limed mud is a rather special type

TABLE I

Rheological Properties of a Lime Base Mud Series

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	API Marsh Funnel viscosity, sec	Gel strength, g	
				Init.	10 min
1·1	11	5·0	32	0	0
1·2	12	5·0	32	0	0
1·3	13	5·0	32	0	0
1·4	14	5·0	32	0	0
1·5	15	5·0	32	0	1
1·6	16	5·5	33	0	1
1·7	18	6·0	33	1	1
1·8	20	7·0	34	1	2
1·9	23·5	8·0	36	1	2
2·0	28	9·5	38	1	3
2·1	34	11·0	41	2	5
2·2	43	14·5	46	2	7
2·3	56	19·5	53	3	9
2·4	80	29·0	64	5	12
2·5	160	43·5	88	10	20

TABLE II

Rheological Properties of Muds from Rig Circulation

Mud type	Sp. gr.	From circulation, ft.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	API Marsh Funnel viscosity, sec	Gel strength, g	
						Init.	10 min
Native shale	1·18	1975	15	9	35	1	3
Lime base	2·10	6980	27	16	39	0	1
Lime base	2·07	7592	34	22	49	0	4

TABLE III

Rheological Properties of Some Low Weight Muds

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	API Marsh Funnel viscosity, sec	Mud type
1·09	23	9·6	108	Caustic treated
1·08	29	12·4	80	Caustic treated
1·09	20	14·5	75	Caustic treated
1·10	27	14·3	80	Caustic treated
1·12	31	20·4	93	Caustic treated
1·09	27	19·8	86	Caustic treated
1·20	29	37·5	180	Caustic quebracho
1·20	29	39·3	195	Caustic quebracho
1·28	7	1·4	—	Natural mud
1·10	7	1·8	35	Natural mud
1·12	13	6·8	43	High pH-caustic-quebracho
1·27	8	1·8	33	Salt water-impermex
1·08	15	5·8	—	Emulsion-gel oil
1·20	17	27·2	58	Gel-chemical
1·19	7	29·3	50	Gel-chemical
1·08	12	13·5	44	Gel-chemical
1·22	11	26·4	48	Gel-chemical
1·20	13	15·2	45	Gel-chemical
1·20	10	29·4	55	Gel-chemical
1·20	12	23·9	51	Gel-chemical
1·20	13	3·5	40	Gel-chemical
1·22	19	24·8	53	Gel-chemical
1·22	14	15·9	46	Gel-chemical

From data in paper by Beck, Nuss, and Dunn.

and wishing to apply the calculations following to more commonly used low-weight muds, the rheological data presented in the paper by Beck, Nuss, and Dunn on the results of field tests on some twenty-four muds have been freely drawn upon. Table III summarizes the properties of these muds.

APPLICATIONS OF EXPERIMENTAL RESULTS

(1) *Circulation Pressures*

Under conditions of turbulent flow, the pressure loss in a pipe is not only directly proportional both to the mud weight and to the square of the mud velocity and inversely proportional to the internal diameter of the pipe, but, in addition, is a function of a friction factor which is usually determined graphically from a knowledge of the Reynolds Number. The Reynolds Number is directly proportional to the mud weight, the mud velocity, and the pipe dimensions and inversely proportional to the plastic viscosity of the mud.

Internal pressure loss in p.s.i/1000 ft pipe

$$= \frac{1{\cdot}296 f W V^2}{D}$$

Reynolds Number (for mud flow)

$$= \frac{396 D W V}{n}$$

where W = mud weight, lb/cu. ft.;
V = velocity of flow, ft/sec;
D = internal diameter of pipe, inches;
n = plastic viscosity of mud, cP;
f = friction factor.

Fig 1 is a plot of lime mud weight *v.* plastic viscosity employing the values given in Table I. Up to

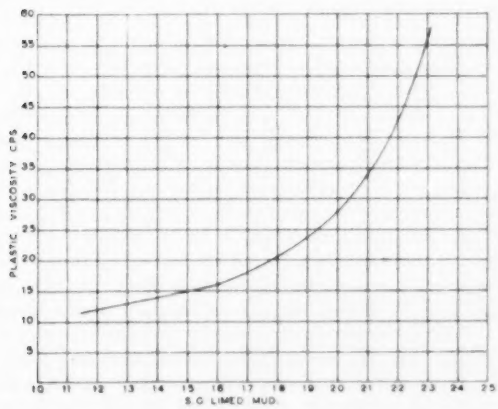

FIG 1

SPECIFIC GRAVITY OF LIMED MUD *V.* PLASTIC VISCOSITY

1·6 sp. gr. the plot is linear, but above this the plastic viscosity increases more rapidly. For a given mud velocity inside a pipe of fixed dimensions, therefore, the Reynolds Number, and hence the friction factor, will remain constant for mud weights up to 1·6 sp. gr. For mud weights above this figure, the value of the Reynolds Number will gradually decrease, increasing f, the friction factor. The resultant pressure drop for

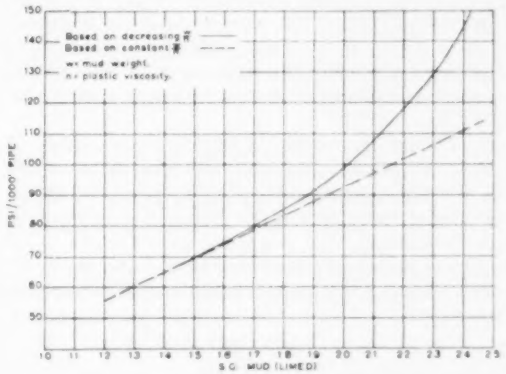

FIG 2

PRESSURE LOSS PER 1000 FT INSIDE 4½-INCH INTERNAL FLUSH DRILL PIPE WITH THROUGHPUT 50 CU. FT. PER MIN

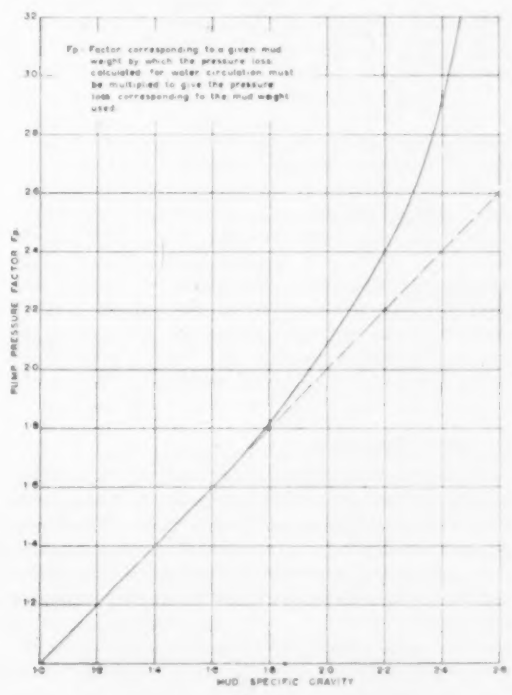
Fp - Factor corresponding to a given mud weight by which the pressure loss calculated for water circulation must be multiplied to give the pressure loss corresponding to the mud weight used.
PUMP PRESSURE FACTOR Fp
MUD SPECIFIC GRAVITY

FIG 3

PUMP PRESSURE FACTOR *V.* MUD WEIGHT

a given length of the pipe for a constant mud velocity will thus be directly proportional to the mud weight up to 1·6 sp. gr., but above this figure the pressure drop will increase more rapidly. Fig 2 is a plot of mud weight *v.* pressure loss/1000 ft of 4½-inch internal flush drill pipe at a throughput of 50 cu. ft/min.

The deviation shown in Fig 2, being dependent only on the decreasing value of mud weight ÷ plastic viscosity, and hence increasing f, is independent of pipe size and mud throughput. It is therefore possible to construct Fig 3, which plots mud weight v. a pump pressure factor Fp. The product of Fp, corresponding to a given mud weight, and the pressure drop calculated for water is the pressure drop that may be expected when circulating mud of weight corresponding to Fp.

Employing the plastic viscosity values given in Table I, the pressure losses inside various sizes of drill pipe for throughputs calculated to produce annulus velocities of 120, 150, and 180 ft/min in various annuli were determined, and are given in Table IV. The practical limitations in terms of mud throughput and pressure losses are more conveniently discussed in the next section.

TABLE IV

Pressure Loss per 1000 ft I.F. Drill Pipe for Various Combinations of Hole Size, Drill Pipe Size, and Mud Throughput

Hole size, inches	Drill pipe size, inches	Annulus area, sq. ft.	Annulus velocity, ft/min	Mud throughput required, cu. ft/min	Internal pressure loss per 1000 ft I.F. drill pipe for mud weights of: 1·20 sp. gr.	2·20 sp. gr.
17½	6⅝ (25·2 lb)	1·4309	180	257	125	255
			150	215	90	185
			120	172	60	125
12	5½ (21·9 lb)	0·6204	180	112	83	165
			150	93	58	120
			120	74	40	79
8½	4½ (16·6 lb)	0·2835	180	51	59	120
			150	42½	43	86
			120	34	29	58
6	3½ (13·3 lb)	0·1295	180	23·2	68	140
			150	19·4	50	104
			120	15·5	32	68

(2) *Critical Velocities*

Laminar flow changes to turbulent flow at a critical velocity, the value of which is a function of the dimensions of the flow system, and the weight and rheological properties of the mud. The formulæ usually employed when calculating the value of the critical velocity of a mud (based on a Reynolds Number of 2000 for laminar flow) is given by :

$$V_c = \frac{8{\cdot}07n + 8{\cdot}07\sqrt{n^2 + 1{\cdot}65WY_tD^2}}{WD}$$

where V_c = critical velocity, ft/sec;
n = plastic viscosity of mud, cP;
W = mud weight, lb/cu. ft.;
Y_t = yield point of mud, lb/100 sq. ft.;
D = internal diameter of pipe in inches *or* four times the hydraulic radius of an annulus in inches, if the flow considered is annular.

Using the rheological data given in Table I, the critical velocity in ft/sec for annular flow in four different hole size–drill pipe combinations was determined for the range of lime mud weights and the results are given in Table V. From an examination of this table it is seen that :

TABLE V

Critical Velocity of Lime Mud in Ft/sec in Various Annuli

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Annulus combination: 17½-inch hole–6⅝-inch D.P.	12-inch hole–5½-inch D.P.	8½-inch hole–4½-inch D.P.	6-inch hole–3½-inch D.P.
			Critical velocity in annulus, ft/sec			
1·1	11	5·0	2·94	3·00	3·16	3·37
1·2	12	5·0	2·80	2·89	3·03	3·25
1·3	13	5·0	2·71	2·80	2·93	3·14
1·4	14	5·0	2·60	2·69	2·79	3·05
1·5	15	5·0	2·51	2·61	2·77	2·97
1·6	16	5·5	2·56	2·65	2·80	3·00
1·7	18	6·0	2·60	2·68	2·84	3·09
1·8	20	7·0	2·70	2·82	2·98	3·22
1·9	23·5	8·0	2·85	2·95	3·02	3·40
2·0	28	9·5	3·02	3·15	3·35	3·57
2·1	34	11·0	3·22	3·36	3·58	3·95
2·2	43	14·5	3·62	3·78	4·08	4·52
2·3	56	19·5	4·13	4·35	4·70	5·27
2·4	80	29·0	4·96	5·27	5·80	6·60
2·5	160	43·5	6·30	6·88	7·95	9·70

TABLE VI

Critical Velocity of Low Weight Mud in 12-inch Hole–5½-inch Drill Pipe Annulus in Ft/sec

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	API Marsh Funnel viscosity, sec	Critical velocity ft/sec in 12-inch hole–5½-inch D.P. annulus
1·09	23	9·6	108	4·33
1·08	29	12·4	80	4·98
1·09	20	14·5	75	5·18
1·10	27	14·3	80	5·26
1·12	31	20·4	93	6·18
1·09	27	19·8	86	6·10
1·20	29	37·5	180	7·81
1·20	29	39·3	195	8·00
1·28	7	1·4	—	1·44
1·10	7	1·8	35	1·82
1·12	13	6·8	43	3·47
1·27	8	1·8	33	1·69
1·08	15	5·8	—	3·31
1·20	17	27·2	58	6·52
1·19	7	29·3	50	6·62
1·08	12	13·5	44	4·84
1·22	11	26·4	48	6·30
1·20	13	15·2	45	4·88
1·20	10	29·4	55	6·63
1·20	12	23·9	51	6·06
1·20	13	3·5	40	2·45
1·22	19	24·8	53	6·18
1·22	14	15·9	46	4·94

Calculated from data given in paper by Beck, Nuss, and Dunn.

(*a*) The minimum annulus velocity necessary to produce turbulent flow is at least 2·5 ft/sec and is generally higher.

(*b*) The critical velocity increases with decreasing annulus size for a mud of given weight and rheological properties.

Table VI shows similar calculations, but for one annulus size only, for lighter muds, using the data given in Table III. Except in the cases of three of the muds, which possessed very low yield values, the critical velocity was usually over 3 ft/sec. In order, therefore, to achieve turbulent flow in the annuli sizes chosen, an ascending mud velocity of at least 150 ft/min is generally necessary, and it should be noted that this velocity, calculated for an "on gauge" hole, for a given mud output, falls appreciably in portions of the hole enlarged by caving and erosion. Table VII illustrates this point.

TABLE VII

Effect of Hole Enlargement on Annulus Velocity

Annulus type		Annulus area, sq. ft.	Annulus velocity in ft/min if hole diameter is increased by :				
Hole size, inches	D.P. size, inches		0 inch	1 inch	2 inches	3 inches	4 inches
17½	6⅝	1·4309	150	132	117	105	94
12	5½	0·6204	150	123	103	88	76
8½	4½	0·2835	150	112	87	70	57
6	3½	0·1295	150	97	69	52	41

Hence under caving hole conditions, even if an annulus velocity of 150 ft/min can be attained to produce turbulent flow conditions (with more efficient mud-carrying capacity), laminar flow will still prevail in enlarged portions of the hole, leading to deposition of drilled and caved debris into the enlargements.

Reference back to Table IV will show the limitations imposed on the attainment of the critical velocity and hence turbulent flow. For example, in the large 17½-inch hole, 6⅝-inch drill pipe annulus, a throughput of at least 215 cu. ft/min is necessary to produce an annular velocity of 150 ft/min, or about twice the output of one Ideal C.350 pump running at 65 strokes/min (18-inch stroke, 7¾-inch liner size, working at 90 per cent volumetric efficiency). A further limitation is imposed on the circulation of such large volumes of mud by pressure loss considerations.

For the four annuli sizes chosen for discussion in this paper, the pressure loss inside the drill pipe calculated for a throughput necessary to produce annulus velocity in the range of 120 to 180 ft/min, represents the following percentage of total pressure drop in the complete circulation system, *i.e.* pump pressure required, for the average depths given. It is assumed that the most efficient circulation system is chosen, involving the correct choice of each piece of circulation equipment.

The higher the annulus velocity and the smaller the annulus area, the higher the percentage of pressure drop contributed by the internal drill pipe loss.

Depth limits, ft	Annulus type	% Pressure drop in D.P. / total pressure drop	
		120 ft/min annulus velocity	180 ft/min annulus velocity
4,000	17½-inch hole–6⅝-inch D.P.	30	40
8,000	12-inch hole–5½-inch D.P.	45	65
12,000	8½-inch hole–4½-inch D.P.	50	65
15,000	6-inch hole–3½-inch D.P.	65	75

Tables VIII and IX are calculations of the approximate total pressure loss to be expected in a circulation system for mud weights of 1·2 and 2·2 sp. gr. respectively. Considering again the 17½-inch hole–6⅝-inch drill pipe annulus, where a mud throughput of 215 cu. ft/min is necessary to produce an annulus velocity of 150 ft/min, the resultant pump pressures at, say, 4000 ft would be: 990 p.s.i. for 1·2 sp. gr. mud; 1844 p.s.i. for 2·2 sp. gr. mud.

The maximum recommended discharge pressure for 7¾-inch liners for a C.350 pump is only 1040 p.s.i. Similar calculations may be carried out for the other annuli sizes, and, in general, it is found that:

(*a*) For low mud weights of the order of 1·6 sp. gr. and less, providing the circulation equipment is carefully selected, an annulus velocity of 150 ft/min can usually be attained, except in the larger hole sizes, without overloading the pumps, even with jet-bit drilling.

(*b*) For mud weights above about 1·6 sp. gr. it is usually impossible to attain this minimum annular velocity unless large pumps are available. In any event, the critical velocity for mud weights above 1·6 sp. gr. is usually well above 150 ft/min.

One very important point is illustrated by reference to Tables VIII and IX, namely, the importance of selecting the most hydraulically efficient circulation equipment. Comparative pressure losses are given for two different drill strings in each hole size and, in addition, the comparative figures for various tool joint types for each drill string, and the effect of varying the drill collar bore.

It should also be remembered that caving hole conditions are very often encountered when penetrating abnormally thick marl successions in which abnormal formation pressures frequently co-exist, necessitating the application of heavy muds. It is safe to say, in this case, that the critical velocity is very seldom attained and the greatest problem is to decide what possible course of action will result in the greatest improvement in hole condition. It is important here to have a thorough understanding of the whole subject of mud-carrying capacity.

Surface Equipment :—
Standpipe 75 ft × 3⅜-inch I.D. pipe (4½ inches)
Hose 75 ft × 3-inch I.D.
Swivel 5 ft × 2½-inch I.D.
Kelley 40 ft × 3½-inch (6⅝ – 5¼ inches), 2⅞ inches (4½ inches), 2¼ inches (3½ – 2⅞ inches)

TABLE
Analysis of Pump Pressures

Hole diameter (inches)			17½								12					
Drill pipe size-weight, inches/lb ft			6⅝/24·7				5½/24·7				5½/24·7			4½/16·6		
Pump throughput, cu. ft/min			257½	215	172	129	270	225	180	135	112	93	74	121	101	81
Annulus velocity, ft/min			180	150	120	90	180	150	120	90	180	150	120	180	150	120
p.s.i./1000 ft of made-up drill pipe		*Tool Joint Type*														
		Internal flush	135	95	64	38	480	345	230	134	95	68	44	287	207	137
		Full hole	143	102	68	40	514	370	245	142	100	72	47	308	220	147
		Slim hole														
		Double str'line														
		Flush joint														
p.s.i./100 ft of drill collar bore		*Bore Drill Collar (inches)*														
		1														
		1¼														
		1½														
		1⅝														
		2														
		2¼														
		2½														
		2¾	580	413	277	163	633	457	300	178	125	90	58	144	103	69
		3	380	270	180	107	415	300	197	116	82	58	38	94	67	45
		3¼	260	185	123	73	284	202	133	78	56	40	26	64	45	30
		3½	180	127	85	50	197	140	93	54	39	28	18	44	32	21
		3¾	128	92	61	36	142	100	66	39						
		4	94	67	45	26	103	74	48	28						
p.s.i. through bit nozzles	Conventional bit with a nozzle fluid velocity of 100 ft/sec	Total nozzle area, sq. in.	6·18	5·15	4·12	3·10	6·50	5·40	4·32	3·25	2·69	2·23	1·78	2·90	2·42	1·95
		Equiv. to 6 nozzles of I.D.	1	1	11/16	11/16	1	1	11/16	11/16	1/2	11/16	1/2	11/16	11/16	11/16
		Equiv. to 3 nozzles of I.D.														
		Equiv. to 1 nozzle of I.D.														
		p.s.i. drop through nozzles	125	125	125	125	125	125	125	125	125	125	125	125	125	125
	Jet bit with a nozzle fluid velocity of 250 ft/sec	Total nozzle area, sq. in.	2·48	2·07	1·65	1·24	2·59	2·16	1·73	1·29	1·08	0·89	0·71	1·16	0·97	0·77
		Equiv. to 3 nozzles of I.D.	1	11/16	5/8	1/2	1	11/16	5/8	1/2	7/16	5/16	5/16	7/16	3/8	5/16
		Equiv. to 2 nozzles of I.D.	1¼	1	13/16	5/8	1¼	1	13/16	5/8	9/16	7/16	3/8	9/16	7/16	3/8
		p.s.i. drop through nozzles	558	558	558	558	558	558	558	558	558	558	558	558	558	558
p.s.i. drop through surface equipment (pump kelley)			515	370	242	142	577	405	266	156	112	80	52	158	115	77
p.s.i. drop through annulus		/100-ft hole–drill collar A/S	—	—	—	—	—	—	—	—	2	1	1	2	1	1
		/1000-ft hole–drill pipe A/S	—	—	—	—	—	—	—	—	7	5	3	6	4	3
Total pump pressure with conventional bits		*Depth (ft)*														
	17½-inch hole with 200 ft of 4-inch bore collars and int. flush drill pipe	1,000	936	705	508	350	1292	954	671	444	(1000 ft)					
		2,000	1071	800	572	388	1772	1299	901	578	(2000 ft)					
		3,000	1206	895	636	426	2252	1644	1131	712	(3000 ft)					
		4,000	1341	990	700	464	2732	1989	1361	846	740	563	409	1526	1134	794
	12-inch hole with 200 ft of 3½-inch bore collars and int. flush drill pipe	5,000									842	636	456	1819	1345	931
		6,000									944	709	503	2112	1556	1074
		7,000									1046	782	550	2405	1767	1214
		8,000									1148	855	597	2698	1978	1354
	8½-inch hole with 200 ft of 2¾-inch bore collars and F.H. (4½ inches) or I.F. (3½ inches)	9,000														
		10,000														
		11,000														
		12,000														
Add 433 p.s.i. to totals if jet bits are to be used	6-inch hole with 200 ft of 2¼-inch collars and int. flush drill pipe	13,000														
		14,000														
		15,000														
	4¾-inch hole with 200 ft of 1¼-inch bore collars and F.J. (2⅜ inches) or slim hole (2¾ inches)	16,000														

VIII

(1·20 *sp. gr. mud*)

8½						6						4½					
	4½/16·6			3½/13·3			3½/13·3			2½/10·4			2½/10·4			2½/6·65	
51	42½	34	59	49	39	23·3	19·4	15·5	27·2	22·7	18·2	8·63	7·20	5·77	11·2	9·35	7·50
180	150	120	180	150	120	180	150	120	180	150	120	180	150	120	180	150	120
58	42	28	370	262	172	66	48	31	292	207	137						
62	44	30	400	282	184	71	51	34							198	140	94
						78	56	36									
						90	65	41				115	83	61			
												181	130	102	300	220	150
												58	40	24	100	71	48
												21	15	10	35	24	16
						61	45	29	82	58	39						
						32	23	15	43	31	20						
						18	13	9	24	17	12						
77	55	37	100	72	48	11	8	5	15	11	7						
46	33	22	61	43	28	7	5	4	9	7	5						
29	21	14	38	27	18												
19	14	10	25	18	12												
13	9	6	17	12	8												
9	7	5	13	9	6												
6	4	3	9	6	4												
1·22	1·02	0·82	1·42	1·17	0·94	0·56	0·47	0·37	0·65	0·54	0·44	0·21	0·17	0·14	0·27	0·23	0·18
125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125
0·49	0·41	0·33	0·58	0·47	0·38	0·22	0·19	0·15	0·26	0·22	0·18						
558	558	558	558	558	558	558	558	558	558	558	558						
33	24	16	68	49	33	16	13	10	18	15	12	—	—	—	—	—	—
5	3	2	6	4	3	5	4	3	7	5	3	—	—	—	—	—	—
13	9	6	10	7	4	14	9	5	11	8	5	18	17	16	15	14	13
(4000 ft)																	
(5000 ft)																	
(6000 ft)																	
(7000 ft)																	
813	611	454	5239	2341	1571	(8,000 ft)											
888	664	490	3619	2610	1747	(9,000 ft)											
963	717	526	3999	2879	1913	(10,000 ft)											
1038	770	562	4379	3148	2089	(11,000 ft)											
1113	823	598	4759	3417	2265	1132	845	584	3780	2718	1844	1814	1389	1085	2840	2088	1487
						1212	902	6.0	4083	2933	1986	1947	1489	1162	3053	2242	1594
						1292	959	656	4386	3148	2128	2080	1589	1239	3266	2396	1701
						1372	1016	692	4689	3363	2270	2213	1689	1316	3479	2550	1808
												2346	1789	1393	3692	2704	1915

Surface Equipment :—
Standpipe 75 ft × 3⅜-inch I.D. pipe (4½ inches)
Hose 75 ft × 3-inch I.D.
Swivel 5 ft × 2½-inch I.D.
Kelley 40 ft × 3½-inch (6⅝ – 5¼ inches), 2⅞ inches (4½ inches), 2¼ inches (3½ – 2⅞ inches)

TABLE
Analysis of Pump Pressures

Hole diameter (inches)			17½								12					
Drill pipe size-weight, inches/lb ft			6⅝/24·7				5½/24·7				5½/24·7			4½/16·6		
Pump throughput, cu. ft/min			257½	215	172	129	270	225	180	135	112	93	74	121	101	81
Annulus velocity, ft/min			180	150	120	90	180	150	120	90	180	150	120	180	150	120
p.s.i./1000 ft of made-up drill pipe		*Tool Joint Type* Internal flush	135	95	64	38	480	345	230	134	95	68	44	287	207	137
		Full hole	143	102	68	40	514	370	245	142	100	72	47	308	220	147
		Slim hole														
		Double str'line														
		Flush joint														
p.s.i./100 ft of drill collar bore		*Bore Drill Collar (inches)* 1														
		1¼														
		1½														
		1¾														
		2														
		2¼														
		2½														
		2¾	580	413	277	163	633	457	300	178	125	90	58	144	103	69
		3	380	270	180	107	415	300	197	116	82	58	38	94	67	45
		3¼	260	185	123	73	284	202	133	78	56	40	26	64	45	30
		3½	180	127	85	50	197	140	93	54	39	28	18	44	32	21
		3¾	128	92	61	36	142	100	66	39						
		4	94	67	45	26	103	74	48	28						
p.s.i. through bit nozzles	Conventional bit with a nozzle fluid velocity of 100 ft/sec	Total nozzle area, sq. in.	6·18	5·15	4·12	3·10	6·50	5·40	4·32	3·25	2·69	2·23	1·78	2·90	2·42	1·95
		Equiv. to 6 nozzles of I.D.	1	1	15/16	13/16	1	1	15/16	13/16	7/8	13/16	3/4	15/16	13/16	25/32
		Equiv. to 3 nozzles of I.D.														
		Equiv. to 1 nozzle of I.D.														
		p.s.i. drop through nozzles	125	125	125	125	125	125	125	125	125	125	125	125	125	125
	Jet bit with a nozzle fluid velocity of 250 ft/sec	Total nozzle area, sq. in.	2·48	2·07	1·65	1·24	2·59	2·16	1·73	1·29	1·08	0·89	0·71	1·16	0·97	0·77
		Equiv. to 3 nozzles of I.D.	1 1/16	15/16	13/16	11/16	1 1/16	15/16	13/16	1/16	7/16	5/16	9/32	7/16	3/8	5/16
		Equiv. to 2 nozzles of I.D.	1¼	1 1/16	15/16	13/16	1 1/16	1 1/16	15/16	13/16	13/32	5/16	1/4	15/32	3/8	19/64
		p.s.i. drop through nozzles	558	558	558	558	558	558	558	558	558	558	558	558	558	558
p.s.i. drop through surface equipment (pump kelley)			515	370	242	142	577	405	266	156	112	80	52	158	115	77
p.s.i. drop through annulus		/100-ft hole–drill collar A/S	—	—	—	—	—	—	—	—	2	1	1	2	1	1
		/1000-ft hole–drill pipe A/S	—	—	—	—	—	—	—	—	7	5	3	6	4	3
Total pump pressure with conventional bits		*Depth (ft)*														
	17½-inch hole with 200 ft of 4-inch bore collars and int. flush drill pipe	1,000	936	705	508	350	1292	954	671	444	(1000 ft)					
		2,000	1071	800	572	388	1772	1299	901	578	(2000 ft)					
		3,000	1206	895	636	426	2252	1644	1131	712	(3000 ft)					
		4,000	1341	990	700	464	2732	1989	1361	846	740	563	409	1526	1134	794
	12-inch hole with 200 ft of 3¼-inch bore collars and int. flush drill pipe	5,000									842	636	456	1819	1345	934
		6,000									944	709	503	2112	1556	1074
		7,000									1046	782	550	2405	1767	1214
		8,000									1148	855	597	2698	1978	1354
	8½-inch hole with 200 ft of 2¾-inch bore collars and F.H. (4½ inches) or I.F. (3½ inches)	9,000														
		10,000														
		11,000														
		12,000														
Add 433 p.s.i. to totals if jet bits are to be used	6-inch hole with 200 ft of 2¼-inch collars and int. flush drill pipe	13,000														
		14,000														
		15,000														
	4¼-inch hole with 200 ft of 1½-inch bore collars and F.J. (2⅜ inches) or slim hole (2⅞ inches)	16,000														

VIII

(1·20 *sp. gr. mud*)

8½						6						4½					
	4½/16·6			3½/13·3			3½/13·3			2¾/10·4			2¾/10·4			2¼/6·65	
51	42½	34	59	49	39	23·3	19·4	15·5	27·2	22·7	18·2	8·63	7·20	5·77	11·2	9·35	7·50
180	150	120	180	150	120	180	150	120	180	150	120	180	150	120	180	150	120
58	42	28	370	262	172	66	48	31	292	207	137						
62	44	30	400	282	184	71	51	34									
						78	56	36							198	140	94
						90	65	41									
												115	83	61			
												181	130	102	300	220	150
												58	40	24	100	71	48
												21	15	10	35	24	16
						61	45	29	82	58	39						
						32	23	15	43	31	20						
77	55	37	100	72	48	18	13	9	24	17	12						
46	33	22	61	43	28	11	8	5	15	11	7						
29	21	14	38	27	18	7	5	4	9	7	5						
19	14	10	25	18	12												
13	9	6	17	12	8												
9	7	5	12	9	6												
6	4	3	9	6	4												
1·22	1·02	0·82	1·42	1·17	0·94	0·56	0·47	0·37	0·65	0·54	0·44	0·21	0·17	0·14	0·27	0·23	0·18
1/32	11/64	11/64	11/32	1/64	15/64	11/64	7/64	11/64	11/32	11/64	7/64	11/32	11/64	11/32	11/32	11/64	11/64
125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125	125
0·19	0·41	0·33	0·58	0·47	0·38	0·22	0·19	0·15	0·26	0·22	0·18						
11/32	11/32	3/32	1/32	11/32	11/32	7/64	7/64	1/64	11/32	11/32	11/32						
7/16	1/2	21/64	15/32	15/32	11/32	1/8	5/64	1/64	13/32	15/32	15/64						
558	558	558	558	558	558	558	558	558	558	558	558						
33	24	16	68	49	33	16	13	10	18	15	12	—	—	—	—	—	—
5	3	2	6	4	3	5	4	3	7	5	3	—	—	—	—	—	—
13	9	6	10	7	4	14	9	5	11	8	5	18	17	16	15	14	13
(4000 ft)																	
(5000 ft)																	
(6000 ft)																	
(7000 ft)																	
813	611	454	3239	2341	1571	(8,000 ft)											
888	664	490	3619	2610	1747	(9,000 ft)											
963	717	526	3999	2879	1913	(10,000 ft)											
1038	770	562	4379	3148	2089	(11,000 ft)											
1113	823	598	4759	3417	2265	1132	845	584	3780	2718	1844	1814	1389	1085	2840	2088	1487
						1212	902	6.0	4083	2933	1986	1917	1489	1162	3053	2242	1594
						1292	959	656	4386	3148	2128	2080	1589	1239	3266	2396	1701
						1372	1016	692	4689	3363	2270	2213	1689	1316	3479	2550	1808
												2346	1789	1393	3692	2704	1915

Surface Equipment :—
Standpipe 75 ft × $3\frac{1}{16}$-inch I.D. pipe ($4\frac{1}{2}$ inches)
Hose 75 ft × 3-inch I.D.
Swivel 5 ft × $2\frac{1}{2}$-inch I.D.
Kelley 40 ft × $3\frac{1}{2}$ inch ($6\frac{1}{4}$ – $5\frac{1}{4}$ inches), $2\frac{7}{8}$ inches ($4\frac{1}{2}$ inches), $2\frac{1}{4}$ inches ($3\frac{1}{8}$ – $2\frac{3}{4}$)

TABLE
Analysis of Pump Pressures

Hole diameter (inches)			17½								12					
Drill pipe size–weight, inches/lb/ft			6⅝/24·7				5½/24·7				5½/24·7			4½/16·6		
Pump throughput, cu. ft/min			257½	215	172	129	270	225	180	135	112	93	74	121	101	81
Annulus velocity, ft/min			180	150	120	90	180	150	120	90	180	150	120	180	150	120
p.s.i./1000 ft of made-up drill pipe		*Tool Joint Type* Internal flush	250	176	117	70	890	640	425	247	177	125	82	527	382	253
		Full hole	265	188	126	74	950	680	450	263	188	133	88	562	408	270
		Slim hole														
		Double str'line														
		Flush joint														
p.s.i./100 ft of drill collar bore		*Bore Drill Collar* (inches) 1														
		1½														
		1¾														
		1⅞														
		2														
		2¼														
		2½	1070	762	502	302	1170	850	550	330	233	165	107	264	190	127
		3	700	500	335	197	770	550	365	213	150	108	70	172	124	83
		3½	475	342	224	134	525	375	247	144	102	74	47	118	84	56
		3¾	330	233	157	93	365	263	172	100	71	51	33	82	59	39
		3½	235	170	112	66	260	186	123	72						
		4	172	123	83	48	190	137	90	52						
p.s.i. through bit nozzles	Conventional bit with a nozzle fluid velocity of 100 ft/sec	Total nozzle area, sq. in.	6·18	5·15	4·12	3·10	6·50	5·40	4·32	3·25	2·69	2·23	1·78	2·90	2·42	1·95
		Equiv. to 6 nozzles of I.D.	1	1	11/16	11/16	1	1	11/16	11/16	1/2	7/16	3/8	7/16	7/16	3/8
		Equiv. to 3 nozzles of I.D.														
		Equiv. to 1 nozzle of I.D.														
		p.s.i. drop through nozzles	229	229	229	229	229	229	229	229	229	229	229	229	229	229
	Jet bit with a nozzle fluid velocity of 250 ft/sec	Total nozzle area, sq. in.	2·48	2·07	1·65	1·24	2·59	2·16	1·73	1·29	1·08	0·89	0·71	1·16	0·97	0·77
		Equiv. to 3 nozzles of I.D.	1	15/16	13/16	11/16	1	15/16	13/16	11/16	7/16	3/8	5/16	7/16	7/16	5/16
		Equiv. to 3 nozzles of I.D.	1¼	1 1/16	1	13/16	1 1/16	1 1/16	1 1/16	13/16	1/2	7/16	7/16	1/2	1/2	7/16
		p.s.i. drop through nozzles	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025
p.s.i. drop through surface equipment (pump kelley)			970	700	460	268	1065	744	497	287	206	148	97	290	208	140
p.s.i. drop through annulus		/100 ft hole–drill collar A/S	—	—	—	—	—	—	—	—	4	2	2	4	2	2
		/1000 ft hole–drill pipe A/S	—	—	—	—	—	—	—	—	13	9	6	11	7	6
Total pump pressure with conventional bits		*Depth (ft)*														
	17½-inch hole with 200 ft of 4-inch bore collars and int. flush drill pipe	1,000	1743	1316	949	649	2387	1759	1246	818	(1000 ft)					
		2,000	1993	1492	1066	719	3277	2399	1671	1065	(2000 ft)					
		3,000	2243	1668	1183	789	4167	3039	2096	1312	(3000 ft)					
		4,000	2493	1844	1300	859	5057	3679	2521	1559	1368	1038	759	2805	2089	1470
	12-inch hole with 200 ft of 3½-inch bore collars and int. flush drill pipe	5,000									1558	1172	847	3343	2478	1729
		6,000									1748	1306	935	3881	2867	1988
		7,000									1938	1440	1023	4419	3256	2247
		8,000									2128	1574	1111	4957	3645	2506
	8½-inch hole with 200 ft of 2¾-inch bore collars and F.H. (4½ inches) or I.F. (3½ inches)	9,000														
		10,000														
		11,000														
		12,000														
Add 796 p.s.i. to totals if jet bits are to be used	6-inch hole with 200 ft of 2½-inch bore collars and int. flush drill pipe	13,000														
		14,000														
		15,000														
	4¾-inch hole with 200 ft of 1½-inch bore collars and F.J. (2¾ inches) or slim hole (2⅜ inches)	16,000														

IX

(2·20 *sp. gr. mud*)

8½						6						4½					
51	4½/16·6 42½	34	59	3½/13·3 49	39	23·3	3½/13·3 19·4	15·5	27·2	2½/10·4 22·7	18·2	8·63	2½/10·4 7·20	5·77	11·2	2½/6·65 9·25	7·50
180	150	120	180	150	120	180	150	120	180	150	120	180	150	120	180	150	120
108	77	52	690	490	320	125	89	58	537	380	262						
116	82	54½	740	525	342	131	94	63							370	263	175
						145	104	72									
						167	119	84				214	155	114			
												336	242	192	550	400	275
												110	76	44	186	133	89
												39	28	19	84	53	30
						113	83	54	151	108	72						
						60	43	28	80	57	37						
142	102	68	187	133	88	34	24	16	46	32	21						
85	61	40	113	80	52	20	15	10	27	19	13						
54	39	26	71	50	33	13	9	6	17	12	8						
35	25	17	46	33	22												
24	17	11	32	23	15												
17	12	8	22	16	10												
12	9	6	16	11	7												
1·22	1·02	0·82	1·42	1·17	0·94	0·56	0·47	0·37	0·65	0·54	0·44	0·21	0·17	0·14	0·27	0·23	0·18
229	229	229	229	229	229	229	229	229	229	229	229	229	229	229	229	229	229
0·49	0·41	0·33	0·58	0·47	0·38	0·22	0·19	0·15	0·26	0·22	0·18						
1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025	1025						
60	44	29	126	90	59	25	20	15	28	23	18	—	—	—	—	—	—
9	6	4	11	8	6	9	6	3	13	9	6	—	—	—	—	—	—
24	17	11	18	13	8	26	17	9	20	14	9	37	34	32	30	38	26
(4000 ft)																	
(5000 ft)																	
(6000 ft)																	
(7000 ft)																	
1507	1135	829	6039	4356	2918	(8,000 ft)											
1647	1234	895	6747	4859	3246												
1787	1333	960	7455	5362	3574	(9,000 ft)											
1927	1432	1026	8163	5865	3902	(10,000 ft)											
						(11,000 ft)											
2067	1531	1091	8871	6368	4230	2097	1560	1072	6939	4979	3377	3418	2616	2047	5231	3931	2789
						2246	1666	1139	7496	5373	3638	3669	2805	2193	5731	4222	2990
						2395	1772	1206	8053	5767	3899	3920	2994	2339	6131	4513	3191
						2544	1878	1273	8610	6161	4160	4171	3183	2485	6531	4804	3393
												4422	3372	2631	6931	5095	3593

(3) *Carrying Capacity of Mud*

Several investigators have examined the factors involved in the transportation of cuttings up the well annulus and proposed modified forms of Stokes's and Rittinger's formulæ (for viscous and turbulent flow respectively) to account for the deviations noticed between calculated and observed rates of transportation. The correction factors applied take into account the flattish shape of the average drilled cutting and the ratio of particle diameter to hole diameter. Two of the most recent papers [4, 5] on the subject of carrying capacity describe experiments in which plastic and aluminium cuttings (the density, shape, and sizes of which were made to agree closely with the observed values for average cuttings) were introduced into the mud annulus and the rates of rise observed under different flow conditions and in the presence of muds of varying composition and rheological properties. The experiments were, however, limited to the use of particle sizes approximating to actual drilled cuttings. Caved fragments are usually greater in size than drilled cuttings, which seldom exceed ½ inch in average diameter and are usually smaller than this. Caved fragments as large as 4 inches or more in diameter have been observed in the field.

In order to observe the effect on slip velocity of increasing the particle size, a range of sizes from approximately ¼- to 4-inch average diameter was considered. The ratio of particle thickness to average diameter was considered constant at 1 : 4.

Employing the rheological data given in Tables I and III for the limed mud series and low gravity mud series respectively, the slip velocities for particles of size range quoted above were calculated for both turbulent and laminar flow conditions. Turbulent slip was calculated using the formulæ given in the Williams and Bruce paper and also the formulæ given in the paper by Hall, Thompson, and Nuss, for comparison purposes. For laminar slip calculations, the formulæ given in the latter paper were used. Tables X–XVI summarize the results of the calculations.

TABLE X

Flatwise Slip Velocity (uncorrected for "edge" effect) of Particles in Turbulent Flow Conditions

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole–5½-inch drill pipe, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·1	11	5·0	3·00	0·66	0·93	1·30	1·86	2·26	2·62
1·2	12	5·0	2·89	0·61	0·86	1·22	1·72	2·10	2·44
1·3	13	5·0	2·80	0·56	0·80	1·13	1·60	1·96	2·26
1·4	14	5·0	2·69	0·52	0·74	1·05	1·48	1·81	2·10
1·5	15	5·0	2·61	0·49	0·69	0·97	1·38	1·67	1·94
1·6	16	5·5	2·65	0·45	0·64	0·90	1·28	1·55	1·80
1·7	18	6·0	2·68	0·42	0·59	0·83	1·18	1·44	1·66
1·8	20	7·0	2·82	0·39	0·54	0·77	1·08	1·33	1·54
1·9	23·5	8·0	2·95	0·35	0·50	0·70	1·00	1·22	1·40
2·0	28	9·5	3·15	0·32	0·45	0·64	0·90	1·11	1·28
2·1	34	11·0	3·36	0·29	0·41	0·58	0·82	1·00	1·16
2·2	43	14·5	3·78	0·26	0·38	0·52	0·72	0·89	1·04
2·3	56	19·5	4·35	0·23	0·32	0·46	0·64	0·79	0·92
2·4	80	29·0	5·27	0·19	0·27	0·38	0·54	0·66	0·76
2·5	160	43·5	6·88	0·15	0·22	0·31	0·44	0·53	0·62

Formulæ after Williams and Bruce

$$V_s = 1{\cdot}35\left(\frac{t}{d}\right)^{\frac{1}{2}} \cdot \left[gd\left(\frac{\rho_s - \rho_m}{\rho_m}\right)\right]^{\frac{1}{2}}$$

for $\frac{t}{d} = \frac{1}{4}$

where: V_s = flatwise slip velocity, ft/sec;
t = thickness of particle, ft;
d = diameter of particle, ft;
ρ_m = mud density;
ρ_s = particle density.

TABLE XI

Slip Velocity of Particles in Turbulent Flow Conditions

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole–5½-inch drill pipe, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·1	11	5·0	3·00	0·42	0·65	0·99	1·60	2·13	2·47
1·2	12	5·0	2·89	0·39	0·59	0·93	1·49	1·98	2·33
1·3	13	5·0	2·80	0·35	0·56	0·88	1·37	1·75	2·18
1·4	14	5·0	2·69	0·33	0·52	0·82	1·26	1·66	2·04
1·5	15	5·0	2·61	0·30	0·48	0·76	1·17	1·56	1·89
1·6	16	5·5	2·65	0·28	0·43	0·70	1·08	1·40	1·76
1·7	18	6·0	2·68	0·25	0·40	0·62	0·99	1·28	1·55
1·8	20	7·0	2·82	0·22	0·35	0·57	0·91	1·17	1·43
1·9	23·5	8·0	2·95	0·20	0·32	0·51	0·80	1·07	1·32
2·0	28	9·5	3·15	0·18	0·28	0·45	0·69	0·95	1·12
2·1	34	11·0	3·36	0·16	0·26	0·40	0·61	0·85	1·00
2·2	43	14·5	3·78	0·14	0·22	0·34	0·54	0·69	0·86
2·3	56	19·5	4·35	0·11	0·18	0·30	0·45	0·57	0·71
2·4	80	29·0	5·27	0·09	0·14	0·24	0·36	0·48	0·59
2·5	160	43·5	6·88	0·07	0·11	0·16	0·28	0·35	0·43

Formulæ after Hall, Thompson, and Nuss

$$V_s = \frac{n \cdot Re}{3 \cdot 2 De \rho_m}$$

for $\frac{t}{d} = \frac{1}{4}$

where: V_s = slip velocity, ft/sec;
Re = Reynolds Number;
De = equivalent particle diameter, ft;
ρ_m = mud density, lb/cu. ft.

TABLE XII

Slip Velocity of Flat Particles Under Laminar Flow Conditions

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole–5½-inch drill pipe, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·1	11	5·0	3·00	0·10	0·46	2·00	8·35	19·00	34·00
1·2	12	5·0	2·89	0·08	0·39	1·70	7·10	16·20	29·00
1·3	13	5·0	2·80	0·07	0·34	1·48	6·20	14·20	25·30
1·4	14	5·0	2·69	0·06	0·29	1·28	5·40	12·30	22·00
1·5	15	5·0	2·61	0·05	0·25	1·10	4·60	10·60	19·00
1·6	16	5·5	2·65	0·03	0·18	0·84	3·60	8·20	14·70
1·7	18	6·0	2·68	0·02	0·13	0·64	2·76	6·40	11·40
1·8	20	7·0	2·82	0·01	0·09	0·45	2·00	4·60	8·40
1·9	23·5	8·0	2·95	—	0·05	0·29	1·33	3·10	5·70
2·0	28	9·5	3·15	—	0·02	0·16	0·81	1·92	3·50
2·1	34	11·0	3·36	—	0·01	0·09	0·47	1·16	2·15
2·2	43	14·5	3·78	—	—	0·03	0·21	0·56	1·06
2·3	56	19·5	4·35	—	—	—	0·06	0·19	0·40
2·4	80	29·0	5·27	—	—	—	—	0·03	0·08
2·5	160	43·5	6·88	—	—	—	—	—	—

Formulæ after Hall, Thompson, and Nuss

$$V_s = \frac{3 \cdot \pi \cdot g}{64 \cdot X \cdot n}\left[td(\rho_s - \rho_m) - 2Y_t(d + 2t)\right]$$

for $\frac{t}{d} = \frac{1}{4}$

where: n = plastic viscosity, lb/sec. ft;
Y_t = yield value, lb/sq. ft.;
d = particle diameter, ft;
t = particle thickness, ft;
ρ_m = mud density, lb/cu. ft.;
ρ_s = particle density, lb/cu. ft.;
V_s = slip velocity, ft/sec;
X = empirical slip velocity factor for laminar slip (dimensionless).

TABLE XIII

Flatwise Slip Velocity (uncorrected for "edge" effect) of Particles in Turbulent Flow Conditions for Low Weight Mud Series

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole-5½-inch drill pipe, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·09	23	9·6	4·33	0·66	0·94	1·33	1·88	2·30	2·66
1·08	29	12·4	4·98	0·67	0·95	1·34	1·90	2·33	2·69
1·09	20	14·5	5·18	0·66	0·94	1·33	1·88	2·30	2·66
1·10	27	14·3	5·26	0·66	0·93	1·31	1·86	2·27	2·62
1·12	31	20·4	6·18	0·65	0·92	1·30	1·83	2·24	2·59
1·09	27	19·8	6·10	0·66	0·94	1·33	1·88	2·30	2·66
1·20	29	37·5	7·81	0·61	0·86	1·22	1·72	2·12	2·44
1·20	29	39·3	8·00	0·61	0·86	1·22	1·72	2·12	2·44
1·28	7	1·4	1·44	0·58	0·81	1·15	1·63	1·99	2·30
1·10	7	1·8	1·82	0·66	0·93	1·31	1·86	2·27	2·62
1·12	13	6·8	3·47	0·65	0·92	1·30	1·83	2·24	2·60
1·27	8	1·8	1·69	0·58	0·82	1·16	1·64	2·01	2·33
1·08	15	5·8	3·31	0·67	0·95	1·34	1·90	2·33	2·69
1·20	17	27·2	6·52	0·61	0·86	1·22	1·72	2·12	2·44
1·19	7	29·3	6·62	0·62	0·87	1·23	1·74	2·14	2·47
1·08	12	13·5	4·84	0·67	0·95	1·34	1·90	2·33	2·69
1·22	11	26·4	6·30	0·60	0·85	1·21	1·71	2·09	2·42
1·20	13	15·2	4·88	0·61	0·86	1·22	1·72	2·12	2·44
1·20	10	29·4	6·63	0·61	0·86	1·22	1·72	2·12	2·44
1·20	12	23·9	6·06	0·61	0·86	1·22	1·72	2·12	2·44
1·20	13	3·5	2·45	0·61	0·86	1·22	1·72	2·12	2·44
1·22	19	24·8	6·18	0·60	0·85	2·21	1·71	2·09	2·42
1·22	14	15·9	4·94	0·60	0·85	1·21	1·71	2·09	2·42

Formula after Williams and Bruce

$$\text{for } \frac{\delta}{d} = \frac{1}{8}$$

TABLE XIV

Correction for "Edge" Effect for Particle Slip under Turbulent Flow Conditions

Hole-drill pipe combination	Hydraulic radius of annulus, ft	Width of annulus ring, ft	Particle diameter, ft					
			0·02	0·04	0·08	0·16	0·24	0·32
			Slip velocity, ft/sec					
Uncorrected figures for 1st mud, Table XIII			0·66	0·94	1·33	1·88	2·30	2·66
17½-in/6⅝-in	0·9062	0·450	0·65	0·90	1·22	1·60	1·82	1·96
12-in/5½-in	0·5417	0·270	0·64	0·87	1·16	1·45	1·60	1·67
8½-in/4½-in	0·3333	0·166	0·62	0·84	1·07	1·27	1·34	1·35
6-in/3½-in	0·2083	0·104	0·60	0·79	0·96	1·06	1·07	1·05

Note:—The figures enclosed by thick line are theoretical only as size of annulus is smaller than particle diameter.

Formulæ after Williams and Bruce

$$V_{s'} = \frac{V_s}{1 + d/D}$$

where: V_s = uncorrected slip velocity, ft/sec;
$V_{s'}$ = corrected slip velocity, ft/sec;
d = particle diameter, ft;
D = equivalent diameter of annulus *or* 4 × hydraulic radius.

Lime Mud Series

From an examination of Tables X, XI, and XII it is seen that:

(*a*) The flatwise slip velocities calculated for turbulent flow conditions using either formulæ (Williams and Bruce, or Hall, Thompson, and Nuss) agree well.

(*b*) The slip velocity for turbulent flow conditions is less than the critical velocity even when the particles are as large as 0·32 ft in diameter.

(*c*) The slip velocity for laminar flow conditions for the smaller particles, approximating to drilled cutting sizes, is less than for turbulent flow conditions.

(*d*) The slip velocity for laminar flow conditions for the larger particles is much greater than for turbulent flow conditions for the lower mud weights; the larger the particle, the higher the mud weight required to produce a laminar slip velocity equal to the turbulent slip velocity.

TABLE XV

Slip Velocity of Particles under Turbulent Flow Conditions for Low Weight Mud Series

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole-5½-inch drill pipe annulus, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·09	23	9·6	4·33	0·39	0·62	0·97	1·55	1·99	2·43
1·08	29	12·4	4·98	0·37	0·62	0·96	1·51	1·97	2·38
1·09	20	14·5	5·18	0·39	0·63	0·98	1·54	2·02	2·44
1·10	27	14·3	5·26	0·37	0·61	0·97	1·43	1·96	2·31
1·12	31	20·4	6·18	0·36	0·57	0·91	1·39	1·92	2·28
1·09	27	19·8	6·10	0·38	0·62	0·97	1·44	1·98	2·32
1·20	29	37·5	7·81	0·34	0·55	0·88	1·35	1·81	2·17
1·20	29	39·3	8·00	0·34	0·55	0·88	1·35	1·81	2·17
1·28	7	1·4	1·44	0·37	0·60	0·94	1·48	1·98	2·42
1·10	7	1·8	1·82	0·43	0·69	1·08	1·67	2·25	2·72
1·12	13	6·8	3·47	0·41	0·64	1·02	1·56	2·05	2·48
1·27	8	1·8	1·69	0·38	0·61	0·96	1·47	1·98	2·32
1·08	15	5·8	3·31	0·41	0·66	1·03	1·65	2·15	2·58
1·20	17	27·2	6·52	0·38	0·58	0·94	1·47	1·92	2·37
1·19	7	29·3	6·62	0·42	0·65	1·02	1·58	2·10	2·58
1·08	12	13·5	4·84	0·42	0·63	1·09	1·64	2·15	2·64
1·22	11	26·4	6·30	0·38	0·60	0·93	1·50	2·00	2·35
1·20	13	15·2	4·88	0·37	0·59	0·96	1·47	1·94	2·31
1·20	10	29·4	6·63	0·40	0·64	0·98	1·46	2·02	2·45
1·20	12	23·9	6·06	0·39	0·60	0·95	1·49	1·97	2·32
1·20	13	3·5	2·45	0·37	0·59	0·93	1·49	1·95	2·34
1·22	19	24·8	6·18	0·36	0·58	0·91	1·42	1·88	2·22
1·22	14	15·9	4·94	0·38	0·59	0·96	1·44	1·88	2·33

Formulæ after Hall, Howard, and Nuss

TABLE XVI

Slip Velocity of Flat Particles under Laminar Flow Conditions for Low Weight Mud Series

Sp. gr.	Plastic viscosity, cP	Yield value, lb/100 sq. ft.	Critical velocity in 12-inch hole-5½-inch drill pipe annulus, ft/sec	Particle diameter, ft					
				0·02	0·04	0·08	0·16	0·24	0·32
				Slip velocity, ft/sec					
1·09	23	9·6	4·33	0·02	0·12	0·56	2·42	5·60	10·1
1·08	29	12·4	4·98	0·02	0·07	0·36	1·58	3·67	6·62
1·09	20	14·5	5·18	0·01	0·07	0·41	1·85	4·33	7·82
1·10	27	14·3	5·26	—	0·06	0·30	1·38	3·23	5·86
1·12	31	20·4	6·18	—	0·02	0·16	0·79	1·87	3·42
1·09	27	19·8	6·10	—	0·03	0·20	0·95	2·26	4·11
1·20	29	37·5	7·81	—	—	0·04	0·30	0·77	1·46
1·20	29	39·3	8·00	—	—	0·04	0·28	0·72	1·36
1·28	7	1·4	1·44	0·26	1·10	4·51	18·3	41·3	74·0
1·10	7	1·8	1·82	0·27	1·12	4·62	18·7	42·5	75·6
1·12	13	6·8	3·47	0·06	0·30	1·33	5·63	12·9	23·1
1·27	8	1·8	1·69	0·21	0·91	3·75	15·2	34·3	61·4
1·08	15	5·8	3·31	0·06	0·31	1·35	5·66	12·9	23·1
1·20	17	27·2	6·52	—	0·01	0·17	0·95	2·33	4·33
1·19	7	29·3	6·62	—	0·01	0·36	2·06	5·10	9·47
1·08	12	13·5	4·84	0·01	0·14	0·75	3·38	7·85	14·2
1·22	11	26·4	6·30	—	0·02	0·26	1·41	3·44	6·40
1·20	13	15·2	4·88	—	0·09	0·52	2·41	5·64	10·3
1·20	10	29·4	6·63	—	0·01	0·25	1·41	3·49	6·50
1·20	12	23·9	6·06	—	0·03	0·30	1·54	3·75	6·9
1·20	13	3·5	2·45	0·01	0·47	1·98	8·16	18·6	33·0
1·22	19	24·8	6·18	—	0·01	0·17	0·90	2·19	4·03
1·22	14	15·9	4·94	—	0·08	0·46	2·13	5·04	9·2

Formulæ after Hall, Thompson, and Nuss

Low Gravity Mud Series

From an examination of Tables XIII, XV, and XVI it is seen that:

(*a*) The flatwise slip velocities calculated for turbulent flow conditions using either formulæ agree well.

(*b*) The slip velocity for turbulent flow conditions, except for three isolated cases, is less than the critical velocity even when the particle is as large as 0·32 ft in diameter.

(*c*) The slip velocity for laminar flow conditions for the smallest particles (0·02 to 0·04 ft diameter) is less than for turbulent flow conditions.

(*d*) The slip velocity for laminar flow conditions for the larger particles is greater than for turbulent flow conditions and is usually greater than the critical velocity.

Table XIV illustrates the effect of correcting the slip velocity for turbulent flow conditions (Williams and Bruce formulæ) for the " edge " effect introduced by varying the hole size–drill pipe combination.

GENERAL CONCLUSIONS

From an examination of the calculations presented, it is apparent that the removal of drilled cuttings, up to about ½ inch average diameter, presents no problem under either turbulent or laminar flow conditions for any mud weight as long as a minimum annulus velocity of about one ft/sec is maintained. The importance of ensuring that this minimum velocity operates at all points in the hole cannot be too strongly emphasized, and the annulus velocity should be adjusted for the greatest " over gauge " portion of the hole.

For larger cuttings and, in particular, caved material, turbulent flow appears to be essential for low gravity muds, and an annular velocity of 150 to 180 ft/min can be expected to maintain a clean hole, providing the critical velocity is below this limit. Unless the critical velocity can be exceeded, laminar flow conditions prevail, resulting in an aggravated hole condition, since the largest caved particles will not be removed from the annulus. A partial remedy would appear to be an increase in the yield value of the mud, but whilst this may result in the removal of some of the larger particles, the tendency to deposit caved material into hole enlargements will be increased, due to the formation of a greater velocity differential across the annulus.

If laminar flow has to be accepted from limitations in pump pressure required to produce turbulent conditions, then it would appear advisable to minimize the velocity gradient across the annulus by keeping the yield value and plastic viscosity of the mud to minimum values, and increase the mud weight, rather than increase the viscosity.

The most noticeable feature in these slip velocity calculations is the overriding effect of increased mud weight. At mud weights above 2·0 sp. gr., although the critical velocity is not usually attainable in practice, the slip velocity of even the largest particles under laminar flow conditions is no greater than that under turbulent conditions.

In the event of caving difficulties being encountered which do not respond to alteration in drilling technique or mud type, *i.e.* conditions which are functions of formation structure, the following remedies would appear to offer the best possible chances of maintaining a clean hole:

(1) Maintain a low viscosity to keep the critical velocity as low as possible.

(2) If pump capacities and pressure limitations permit, circulate the annulus at a rate higher than the critical velocity to encourage turbulent flow pattern.

(3) Increase mud weight within the limits allowed by the critical velocity and pump limitations whilst still maintaining turbulent flow.

(4) If pump limitations and hole size preclude the establishment of turbulent flow conditions, and this is invariably the case when penetrating abnormally pressured marl formation at depth with heavy muds, circulate, at the maximum possible rate, a mud of minimum viscosity at a mud weight which satisfies possible limitations applied by pump pressure and formation loss considerations. In this connexion the importance of drill pipe rotation should be emphasized, since its effect is to reduce channelling of the mud, with subsequent trapping of debris in the quiescent section of the annulus.

Finally, it is worth recording that in recent operations in a deep well,[7] which penetrated over 4000 ft of steeply dipping formation under abnormal pressure conditions, necessitating the application of heavy muds seldom less than 2·1 sp. gr. and frequently as high as 2·30 sp. gr., good recovery of caved debris, frequently 4 inches in diameter, was achieved with low viscosity limed mud, indicating the excellent carrying capacity of this extremely mobile fluid.

ACKNOWLEDGMENTS

The writer wishes to express appreciation to the Chairman of the British Petroleum Company Ltd. for permission to publish the information included in this paper and to the American Petroleum Institute for permission to reproduce Table 2, p. 21, *Drilling and Production Practice*, 1947.

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DISCUSSION

G. P. Laird: Trends in the industry, first to jet drilling, and perhaps later to the turbine, make it most important that one should know more about those mud properties which control flow characteristics. In regard to the turbine, it is considered that Mr Cooke's reference to optimum drill pipe sizes is particularly significant, and it would be very interesting to know more precisely what "optimum sizes" really amount to. From a practical point of view, the conventional rotary system imposes many limitations in this respect, but, with the turbine, the specifications of the drill pipe can be changed very considerably. In fact, it is believed that one of the main reasons behind the Russian use of the turbine was the difficulty in obtaining drill pipe which was strong enough for drilling in the conventional way.

Mr Cooke's paper is a particularly valuable contribution, as it discusses the hydraulics of mud flow in relation to a specific problem, one which has been with us for a very long time, and one which is, in some areas at any rate, a very major one. I refer to the problem of caving hole trouble. The author has shown us that turbulent flow is more efficient than laminar flow in cavings removal, but that as the mud weight increases, it is the mud weight which becomes the overriding factor, rather than the type of flow. This is particularly interesting to me because in Trinidad we have a problem in a long shale section, which we first attempted to drill using a 95 lb/cu. ft. mud, and every time we ran into serious trouble with caving and hole difficulties. These we were able to overcome by raising the weight of the mud up to 115–120 lb/cu. ft., but the use of these excess mud weights presented other difficulties, and were disadvantageous in many ways. In order to try to get away from these difficulties we reverted to the use of 95 lb/cu. ft. mud for one well, but on this occasion conditions were arranged to ensure that turbulent flow took place in the annulus during drilling. However, this approach was unsuccessful, the trouble recurred and persisted, and eventually a string of pipe was lost. It was necessary to go back to the use of heavy mud, and this time it was introduced before the trouble was encountered. It was found that when this was done, either the trouble did not occur at all, or if it did, it was of minor importance.

This experience suggests that whatever the merits of turbulent flow, there are many cases in which it is far better to raise the mud weight at the expense of the other properties of the mud, even though this may preclude the chances of attaining turbulent flow. At the same time, it suggests that raising the weight of the mud is not only a corrective, but is also a preventive, and I should very much like to hear any views on this aspect of the problem.

P. W. Cooke: Answering first your request for more information on optimum drill pipe size, this aspect of mud hydraulics is perhaps best illustrated by considering the internal pressure loss per 1000 ft of drill pipe of various standard sizes. Selecting 150 ft/min as a useful and effective annulus velocity inside a 12¼-inch hole, the actual pressure losses per 1000 ft inside the drill pipe are shown in the Table below.

As would be expected, the pressure losses are inversely proportional to the fifth power of the pipe internal diameter for a given throughput.

It does illustrate that one has to be very careful in selecting the optimum circulation equipment, and this is particularly important when choosing drill pipe and selecting the drill collar bore. When jet bit drilling, in which one aims at attaining a nozzle fluid velocity of 200–250 ft/sec, the pressure loss through the bit is easily determined and remains constant throughout all one's calculations. So one can only minimize the final pressure loss for a given throughput by carefully selecting the drill pipe, surface equipment, drill collar bore, and, in fact, choosing any part of the circulation system to have the maximum bore, within practical limitations. By this means, one is able to concentrate the greatest proportion of available pump hydraulic horse-power at the bit, which is where it is needed.

D.P. size	Internal pressure loss per 1000 ft of pipe at a throughput producing an annulus velocity of 150 ft/min in a 12¼-inch hole, p.s.i.
5½-inch I.F.	58
5½-inch Tubing	50
5-inch Xtra hole	106
4½-inch F.H.	220
4½-inch Tubing	154
4-inch Slim hole	488
4-inch Tubing	308
3½-inch Slim hole	1220
3½-inch Tubing	704

With regard to turbulent flow *v.* mud weight for combating caving, I have tried to distinguish in this paper between abnormal caving, *i.e.* caving which is a function of abnormal formation conditions, and the caving which one can often cure by altering other factors, such as decreasing the fluid loss of the mud, or by changing the drilling practice. Now, when one meets abnormal caving, it is very often accompanied by pressure conditions requiring heavy mud. That is why the attainment of turbulence is seldom achieved and this puts a limitation to the conclusions that I have drawn. I do not think that I can say more except that when one has to apply heavy muds and cannot obtain turbulent flow, then I maintain that one should try to keep the viscosity as low as possible and achieve the maximum throughput.

W. L. Wilkinson: In Newtonian flow in pipes it is well known that the onset of turbulence takes place at a Reynolds Number of about 2000. However, in the flow of non-Newtonian fluids the same criterion may not apply. (There is also the inherent difficulty in the definition of Reynolds Number that the viscosity is not constant but is a function of the rate of shear.) In fact, it is more likely that turbulence does not commence until the Reynolds Number is considerably greater than 2000. In any case there is strong experimental evidence that the transition zone is much wider than for Newtonian flow, *i.e.* the change from laminar to fully developed turbulence does not take place over a narrow range of Reynolds Numbers (*e.g.* 2000–3000 for Newtonian flow) but may extend to Reynolds Numbers in excess of 25,000. In such cases there is the possibility that the flow in certain parts of the system, *e.g.* a pipe, may be turbulent and in others laminar.

It is suggested, therefore, that the calculations on p. 72 which predict the critical velocities for turbulent flow could be misleading, and turbulent conditions in the annulus may be even more difficult to achieve than the paper suggests.

P. W. Cooke: I would agree that in the case of non-Newtonian fluids, a Reynolds Number of 2000 is probably

too low to expect the onset of turbulence. The effect on the critical velocity of increasing the Reynolds Number is shown below:

Pipe I.D. inches	Reynolds Number			
	2000	3000	4000	5000
2	3·40	4·40	5·28	6·12
3	3·16	3·98	4·72	5·41
4	3·04	3·78	4·48	5·10
5	2·98	3·68	4·35	4·90
6	2·88	3·61	4·22	4·80
7	2·87	3·60	4·15	4·72
8	2·82	3·54	4·12	4·65
9	2·80	3·48	4·08	4·62

Critical velocities in ft/sec for 1·2 sp. gr. mud:
n = 12 cP plastic viscosity
Y_t = 5 lb/100 sq. ft. yield value

The figure of 25,000 seems rather high, but I do remember seeing 10,000 suggested as a possible figure at which the change-over from streamline to turbulent flow could be expected. The use of 10,000, or even 25,000 would in any case increase all the critical velocity figures given in Tables V and VI, and would not affect the main argument, namely that whilst turbulent flow is desirable, it is seldom achieved in practice. It is all a matter of degree. It has been the custom in past literature on mud flow to employ Reynolds Number values of 2000 and 3000 when calculating critical velocities. One further point is that with modern low viscosity lime muds of extreme mobility and very little, if any, yield value, the onset of turbulent flow might well occur at quite low values of the Reynolds Number, since the flow characteristics of such muds are almost Newtonian.

Dr E. B. Evans: 2000 is the normal "change-over" value for ordinary liquids. What is the evidence for using 2000 in this particular type of system? Is it just that you are taking the normal change-over value, or is there evidence that it applies to these mud systems?

P. W. Cooke: I agree that there is little, if any, evidence that the figure of 2000 can be directly applied to mud systems. Previous contributors in this field have taken the figure applicable to Newtonian fluids and applied it to muds, since we are more interested in determining relative values rather than actual values of the critical velocities.

Dr E. B. Evans: Then there has not really been enough detailed work on the viscous properties of these suspensions?

P. W. Cooke: I think that is generally true.

N. Healey: While agreeing with the author's calculations and his arguments about the advantage of turbulent flow in the lifting of cuttings and cavings, I think it is the experience of most people who have worked extensively with drilling fluids (and certainly it is common practice in many areas throughout the world, *e.g.* the U.S.A., South America, and Indonesia) that when very serious caving conditions are encountered, one of the first actions taken is to increase the viscosity of the drilling fluid to induce laminar flow. This has regularly been successful in eliminating caving. By this, I do not simply mean a mere efficient removing of cuttings, but an actual stabilization of the hole, so that no further caving occurs. Once the drill pipe is removed from the hole, the latter becomes clean, and no difficulty is experienced on running in again. Hence, what I am interested in is how far the suggestions you mention at the end of the paper for overcoming caving are based on theoretical considerations and to what extent on actual experience.

P. W. Cooke: The paper has a concluding paragraph on recent operations in a deep well, work on which first made me give serious thought to the subject of this paper. There, we did, in fact, have 4000 ft of caving problems, since we were drilling in abnormally pressured marl down a very steeply dipping and frequently vertical flank of the structure. The weight of the mud was between 1·9 and 2·2 sp. gr. throughout the whole of the caving section. Low viscosity, very mobile lime mud was used, and cavings as large as 3–4 inches in diameter were easily recovered at the surface. The mud weight may have been the overriding factor, but if there was no turbulent flow, at least there was a low viscosity, varying from 40 to 60 sec Marsh Funnel for a 2·1 to 2·2 sp. gr. mud. I would not consider 60 sec a high viscosity. There are reports of areas in the U.S.A. and Canada where viscosities as high as 200–300 sec Marsh Funnel have been used. I cannot see the necessity for such high figures, and I have discussed this point with many operators. My feeling is that, usually, these high viscosities result from chemically treating the mud to improve some other factor, such as the fluid loss, and it could well be that any reported hole improvement was not due to the viscosity increase but to the change in some other mud property. Whether or not to increase the viscosity is the controversial part of the paper and one of the very reasons that I wrote it. I have talked to many mud experts, and it is evident that there are two distinct camps, as it were: those who support a high viscosity to alleviate caving difficulty and those who would rather maintain a low viscosity. My views agree with the latter, and I should add that we are probably in the minority.

N. Healey: I would say, with reference to the concluding remarks in the author's reply, that the increases in viscosity were definitely purely increases in viscosity and did not involve any other properties of the drilling fluid. The reduction of filter loss is normally the first thing one would try to improve hole conditions. Next, one would increase viscosity, and finally one would effect an increase in the mud weight. This is tried last, as it is the most expensive method.

From practical experience, I would say that viscosity increase alone can give results in overcoming caving, since the water loss was almost invariably extremely good, both before and after the viscosity change.

G. K. Jones: Could I suggest a reconciliation between the two schools of thought? Drilling muds can be considered as Bingham bodies. Simultaneous decrease of the yield value of the mud and increase of the pumping rate will create turbulent flow, with a flat velocity profile in the annulus, a condition which seems to be well suited to the removal of cavings from the hole. A similar velocity profile can, however, be created by increasing the yield value of the mud to a high level and reducing the pumping rate: under these conditions plug flow will be induced, particularly in enlarged sections of the hole, with the characteristic flat velocity profile encountered in plug flow. It is only in the intermediate zone of moderate mud yield value and pumping rate that a parabolic velocity profile will be encountered, and it may be that this is the one flow region which must be avoided under

caving conditions, in that detritus is not easily brought to the surface, but migrates to the low velocity regions in the annulus, where accumulation creates a potential drilling hazard.

P. W. Cooke: I would agree partly with the remarks of Mr Jones, and I neglected to point out that portion of the curve showing plug flow characteristics. There is the old remedy when one encounters caving difficulties, especially in lost circulation zones, of putting into circulation an extremely viscous plug. Whether, in fact, it travels up the annulus in the form of a plug and has plug flow characteristics, it is not possible to say. This technique is also used on occasion to obtain cutting samples when lost circulation is experienced. If, however, we accept the fact that there is a velocity differential across the annulus with laminar flow conditions, then particles must be transported to the flow edges and left behind. With really thick viscous mud, these particles, after deposition into hole enlargements, are held in position by thick gelled mud and do not thereafter give any trouble. The chances of recovery are certainly reduced under laminar flow conditions, and my calculations were designed to remove them rather than leave them in, since any material left in the hole is a potential hazard at a later stage. I must agree that plug flow could explain the action of high viscosity mud in frequently improving hole conditions when caving problems are encountered.

L. A. Bushe: In dealing with the removal of cavings from the well, the author says that the best place for the cavings is on the surface, and he suggests that if they are not removed from the annulus (between the drill pipe and the wall of the hole) they will remain a hazard. While it is agreed that, generally speaking, it is desirable to remove both drilling detritus and cavings from the mud stream, I disagree with him if he means that unless all such particles are removed they will necessarily constitute a hazard to drilling progress. When caving has taken place and sections of enlarged, or over-size, hole are present I do not think we need be alarmed if particles of the cavings are deposited into these hole enlargements; indeed, I would even suggest that these particles may assist, in conjunction with increased viscosity and weight of the mud (where this is practicable), in sealing off the dangerous portions of the hole. Should such a sealing effect be obtained by these means, it is, of course, important that every care is taken to ensure that sudden and drastic changes are not subsequently made in the weight, viscosity, or velocity of the mud in the annulus which might disturb the stability of the hole and induce further caving of the same section.

In regard to the drilling equipment, Mr Cooke mentions particularly the C.350 pump and shows that under certain conditions of hole and mud this pump does not have the capacity to produce an annulus velocity of 150 ft/min, which is required to obtain turbulent flow. It might appear from the paper that the limitations of the pump are due to the maximum discharge pressure recommended by the makers, whereas it is, of course, possible to increase the pressure above the stated figure of 1040 p.s.i. by fitting smaller liners to the pump and accepting a lower throughput of mud.

Reference is also made in one of the diagrams to the G.7000 pump, which description might lead the uninformed to assume that this pump has twice the capacity, both as to volume and pressure, of the C.350 pump for a similar liner size. This is not the case, owing to the shorter stroke of the larger pump.

For the above reasons it is suggested that it would be clearer if the author always described a pump by its liner diameter and length of stroke (*e.g.* 7¾ inches × 18 inches, 6½ inches × 18 inches, etc.) and not by the manufacturers number of a particular model.

To revert to some of the aspects of drilling muds, members might be interested to hear something about the deepest well in Trinidad, which has been drilled to more than 16,000 ft and with casing cemented at 9000 ft carried over 7000 ft of 8½-inch diameter open hole before completion. A lime–starch oil emulsion mud was used, and below 10,000 ft the mud weight was maintained at 130–135 lb/cu. ft., while considerable variations were made to the viscosity, gel strength, and water loss of the mud.

Although deep drilling in Trinidad is notoriously difficult owing to the frequency of stuck drill pipe, the drilling of this well seems to indicate that by careful mud control and drilling technique this difficulty can be avoided and good drilling progress made, for even below 15,000 ft a penetration rate of 8 ft/hr was obtained with a pump throughput as low as 300 gal/min, the pump pressure being around 1800 p.s.i.

I agree with the author in stressing the importance of drill pipe rotation to reduce channelling of the mud, but would add the caution that there is considerable danger of sticking the pipe if it is rotated for a long period in one spot. As is well known, a rotary-drilled hole is never truly vertical, with the result that the drill pipe bears more or less heavily against the wall of the hole in the numerous irregular sections of the borehole, and if it is merely rotated there is a great danger of " key-seating ", even though free mud circulation may be maintained. I would therefore suggest that vertical movement of the drill pipe at frequent intervals is just as important as rotation, and that this technique should be employed whenever there is a danger of stuck drill pipe, either from caving or from any other cause.

P. W. Cooke: The query on pump capacity is best answered by referring to the following figures, in which pump output is plotted against the maker's recommended maximum pump discharge pressure. This sort of diagram I have found to be most useful when making hydraulic calculations. The pump capacity lines on the diagrams take care of all possible liner size changes. Consider, for example, Fig 4, illustrating the calculated pressure loss in a well with which I was associated, with a 14¾-inch hole at 4000 ft. The first and most obvious point to notice is the great saving in pump pressure that can be made if the circulation equipment (drill pipe size, drill collar bore, etc.) is carefully selected. Thus, for example, to obtain an annulus velocity of 180 ft/min, requiring high throughputs, with the actual equipment on the site, two G.1000 pumps (8½ inches × 16 inches) are hardly sufficient, but with optimum equipment, the resultant pump pressure is well inside the capacity of two C 350 pumps (7¾ inches × 18 inches). The second point which the figure illustrates is the comparison of pump hydraulic horse-power for several pump types. Thus the maximum throughput with the largest liner size at the maker's recommended maximum speed of two G.700 pumps (8 inches × 14 inches) is slightly less than that of two C.350 pumps (7¾ inches × 18 inches), but for the smaller liner sizes the G.700 has about 860 p.s.i. higher pressure rating above a C.350 pump, and this is where a G.700 pump scores over a C.350 pump. Figs 5 and 6 illustrate the calculated pressure loss in an 8½-inch hole at 12,000 ft employing either conventional bits or jet bits for mud weights up to 2·3 sp. gr., at annular velocities varying from 90 to 180 ft/min. Equipment selected for the calculations was 4½-inch surface lines and standpipe, 3½-inch bore hose, 2¾-inch bore kelley, 200 ft

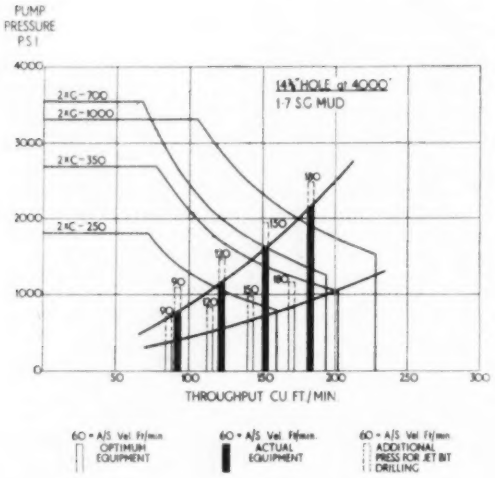

Fig 4

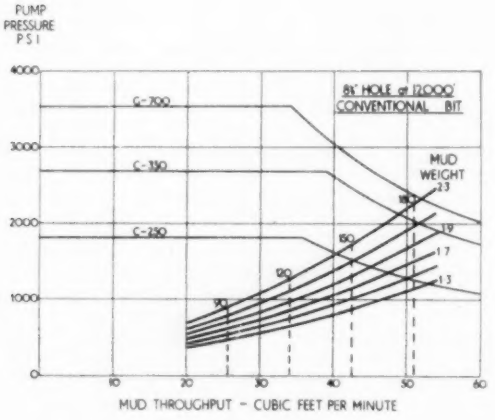

Fig 5

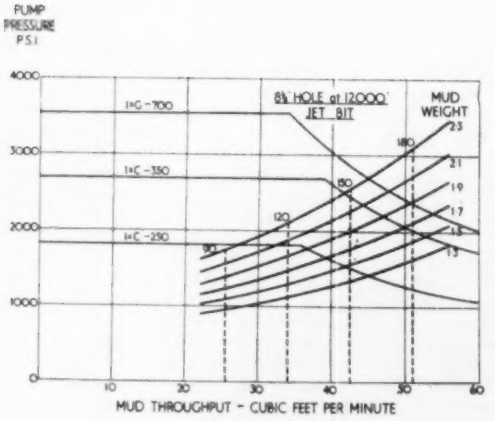

Fig 6

of $2\frac{3}{4}$-inch bore drill collars, and $4\frac{1}{2}$-inch full hole drill pipe. A nozzle fluid velocity of 100 ft/sec was assumed for conventional bits and 250 ft/sec for jet bits.

Finally, I would agree with Mr Bushe about the reciprocal motion of the pipe, but repeat that I do not think the importance of drill pipe rotation to remove debris is sufficiently realized.

E. A. E. Nichols-Roth: I should like to ask the author whether he has any practical suggestions; in the case of caving, near the bottom of the hole, it is not always easy to diagnose immediately whether this is due to geological causes or to some change in drilling practice, whichever may have taken place.

The driller's first reaction is undoubtedly to try to pull his string back above the caving; but, if he does that, he loses the one great advantage which he might have had (according to the author) in leaving the string down below and continuing to rotate, to try to clear the obstruction through his mud flow.

I quite agree that the usual method of retracing the hole is to feed down as carefully as possible, using the mud flow at maximum velocity and minimum viscosity.

If this method, advocated by Mr Cooke, does not meet the case in point, the string will have to be withdrawn and washed down again with quite a different bit—a solid, fish-tail, or chopping bit, for instance; to go through this obstruction by some mechanical means.

Would Mr Cooke suggest dependence on the hydraulics to that extent; to leave the string at the bottom of the hole, continue to rotate, and try to clear the obstruction in the first instance through the mud flow; realizing, however, that in doing so one might lose the chance to pull back before jamming the string?

P. W. Cooke: This is indeed a problem, but one with which drilling personnel are frequently faced. One point to remember is that the clearing of the debris will have to be done eventually and before further drilling progress can be achieved. I am of the opinion that before pulling back, little harm can result in trying a few remedial measures. The problem is relative to the type of caving. Altering, for example, the mud properties at such a moment is frequently not to be recommended and indeed, even if desired, takes time. There would appear to be no harm in achieving the maximum circulation rate to see if the hole condition can be improved. Under abnormal conditions of caving, the natural reaction is to come off bottom frequently, but I can also recall occasions when we persevered on bottom. My experience, since I am not a driller, is probably insufficient to decide between the two methods.

E. A. E. Nichols-Roth: I particularly had in mind exploration drilling, where one does not carry a second string very often.

P. W. Cooke: I would like to pass your comments to someone more versed in actual drilling experience. My opinion would be to stay on bottom and circulate, but this could be hazardous under certain circumstances. Frequently, the technique which I have seen employed is to pull back for safety, and either increase mud viscosity, or alter some other mud property, or indeed, change the mud type and then go down and try again.

The mud engineer can usually only recommend a course of action. Whether drillers accept this advice or not is often dependent on their relative experiences in difficult formations. Perhaps the best answer is to say that individual cases merit individual action.

G. P. Laird: I would like to ask whether the reference to "pulling up" means coming up off bottom for a short distance and maintaining circulation or pulling a number of stands, thereby interrupting circulation.

E. A. E. Nichols-Roth: I mean pulling back past the caved portion of the hole, whilst maintaining circulation, unless, of course, the distance necessitates breaking the string and interrupting circulation.

G. P. Laird: I cannot see that there would be any harm in pulling back a short distance while maintaining circulation. The first indication of trouble occurring might be the appearance of the cavings on the screen, possibly a labouring of the rotary table, and in very extreme cases some pressure rise, accompanied by a reduction in the weight of the string. If the occurrence of caving is severe enough to affect the hydraulic system, there is a danger of the string being pumped up hydraulically, and it may then become necessary to shut down the pump. It is better not to run this risk if it can be avoided by pulling back up. I think that pulling back a short distance is the best course and if, after a circulation cycle, no further caving shows up on the screen, it can then be established that no cavings are coming into the hole except within the last thirty feet or whatever is the distance pulled. Caving can show up in a mud stream, but nobody can be certain that it has necessarily come from the bottom.

Dr E. B. Evans: I should like to thank Mr Cooke for his very interesting and ably presented paper. He has told us that he wrote the paper largely with the object of stimulating a discussion, and in addition to the very excellent presentation of his own views, he has certainly succeeded admirably in doing this.

The meeting then closed with a warm vote of thanks to the author of the paper.

OVERALL AND LIQUID FILM EFFICIENCIES IN A KASKADE COLUMN *

By F. H. GARNER,† O.B.E. (Past-President), S. R. M. ELLIS,† and E. S. LUXON† ‡

SUMMARY

Overall efficiencies in the Kaskade tray are presented for the systems methyl*cyclo*hexane–toluene and methyl–alcohol–water and liquid film efficiencies for the desorption of oxygen from water. At total reflux, the gas film resistance for all these systems decreases with increasing vapour velocity, whereas the liquid film resistance remains relatively constant.

NOMENCLATURE

D_L	Liquid film diffusivity
E_{g0}	Overall gas or vapour efficiency
E_L	Liquid film efficiency
N_L	Number of liquid film transfer units
$N_{Lt°C}$	Number of liquid film transfer units at $t°$ C
$N_{L20°C}$	Number of liquid film transfer units at 20° C
N_G	Number of gas or vapour film transfer units
G	Mass vapour velocity, lb/hr/ft²
V	Linear superficial velocity, ft/sec
L	Liquid rate, lb/hr/ft²
x_{n+1}	Concentration of oxygen in p.p.m. entering the tray
x_n	Concentration of oxygen in p.p.m. leaving the tray
x_n^*	Equilibrium concentration of oxygen in p.p.m. leaving the tray in p.p.m.

INTRODUCTION

THE tendency in recent years to develop new vapour–liquid contacting plates and packings has led to special interest in their performance and limitations. The Kaskade tray was introduced in 1943 [1] as an attempt to overcome the low capacity limitations of the bubble-cap tray. The Kaskade, together with its successor, the Benturi, has been widely adopted in the chemical and petroleum fields, and over 350 commercial installations have been reported.[2]

The competition between these and other new designs, such as the Turbogrid,[3] Kittel tray,[4] Spraypak,[5] Parapak,[6] and the improved designs of the bubble cap and perforated plate makes the selection of the best contacting means for a specific service of utmost importance. This selection can be carried out satisfactorily only when sufficient data on efficiency, flexibility, capacity, pressure drop, and cost are available.

Results have been previously reported for the systems methyl*cyclo*hexane–toluene and ethyl alcohol–water.[7] This work is now extended to higher boil-up rates, and results are reported for the desorption of oxygen from water.

APPARATUS

The apparatus consisted of a six-plate distillation column and a single-plate unit used for the oxygen desorption tests. Identical trays were used in each unit. Details of the tray have been previously reported.[7]

DISTILLATION COLUMN

A flow diagram of the distillation unit is given in Fig 1. The solvent from reboiler (A) passes through the column (B) to the condenser (C), the overheads being returned to the column via the nest of rota-

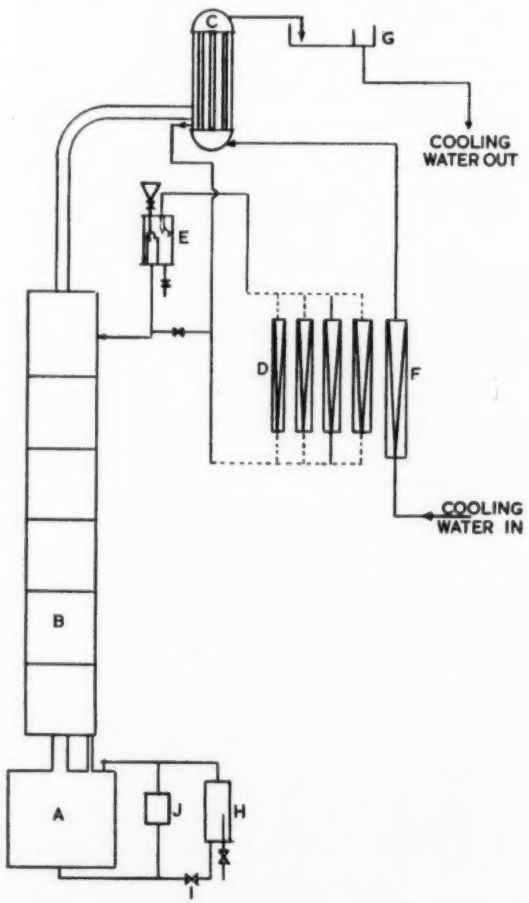

FIG 1

FLOW DIAGRAM OF SIX-PLATE DISTILLATION UNIT

* MS received 19 October 1956.

† Chemical Engineering Department, University of Birmingham.

‡ Now with The Distillers Co. Ltd., Epsom.

meters (D) and a reflux measuring device (E). The cooling water is measured in the rotameter (F), and from the condenser is passed to a drain through a small tank (G), which incorporated a float-controlled electrical cut-out. The reboiler was fitted with a sight glass (H), a magnetic valve (I) being in the connecting line. Another cut-out (J) was controlled by the reboiler level.

Details of the distillation column were as follows :

Number of trays	6
Tray spacing	16 inches
Cross-section of tray	8 inches × 2 ches
Total slot area	2 sq. in.
Downcomer cross-section	2 sq. in.

Experimental Details

The time required to reach equilibrium was ascertained by continuous sampling from the trays of the column until the composition on each tray was constant. The times required to reach equilibrium were approximately 8 hours for the system methyl*cyclo*hexane–toluene and 12 hours for methanol–water.

The vapour-sampling device previously described [7] provided for the continuous condensation of the vapour and the revaporization of the condensate into the column. Samples taken by this recycling method were compared with samples of vapour directly condensed into sampling bottles. Within the accuracy of analysis the samples were identical in composition.

During the experimental runs the lagging and column temperatures were checked to ensure that the lagging heaters were functioning and maintaining adiabatic conditions.

When equilibrium was reached, the pressure drop across each tray was measured, and then sampling was carried out by isolating the sample pots. The samples were then run off into cooled bottles and stored in a refrigerator until required for analysis.

The overheads were then measured by diverting the reflux into a receiver over a known period and weighing. As the reflux was not at its boiling point, a correction was necessary to ascertain the actual vapour velocity.

Single Tray Unit

The layout of the single-plate unit, which was constructed with Perspex walls to allow observation, is given in Fig 2. All flow measurements were by orifice meters, and provision was made for sampling of the inlet and outlet water with continuous circulation through the sampling flasks during runs.

The air was supplied by a blower capable of delivering 3500 ft³/hr against 12-inches W.G. The air entered the base of the plate (A) via a straight length of 2-inch standard iron pipe, an orifice meter (B), and a butterfly control valve (C). From the plate it passed through a cyclone separator (D) to the atmosphere. The cyclone separator served to remove entrainment for return into the bottom reservoir (E).

The water supply was a continuous circuit. A centrifugal pump (F) delivered it to a 10-gallon constant head tank (G) from the reservoir. The level in this tank was maintained by an overflow. The water flowed to the plate via an orifice meter (H) and a control valve (O) and thence back to the reservoir. A small drain pipe (I) prevented any water from getting to the air orifice and blower.

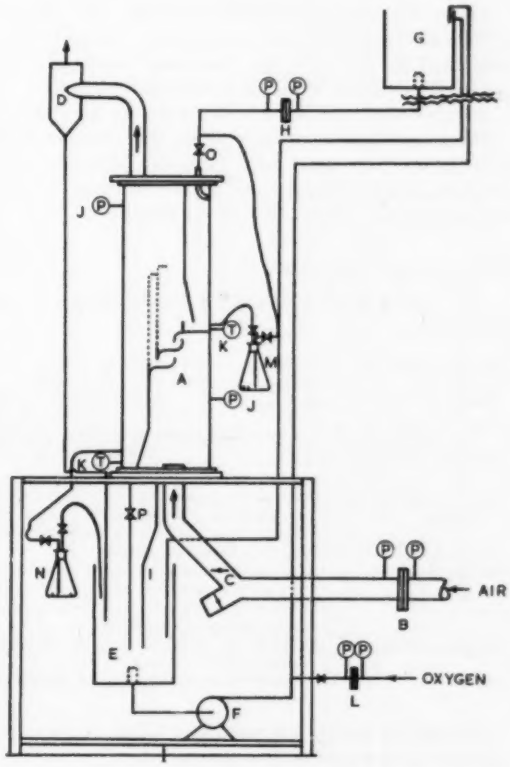

FIG 2
FLOW DIAGRAM OF SINGLE-PLATE UNIT

Pressure tappings (J) were provided to measure the pressure drop across the tray, and a mercury-in-glass thermometer (K) was used to measure the inlet and outlet water temperatures.

The water was enriched with oxygen supplied to the delivery side of the pump from a cylinder via a small orifice meter (L). Provision was made for sampling the inlet and outlet water. The sampling flasks (M) and (N) are shown in position.

Experimental Procedure

The water-circulating pump was switched on and the oxygen introduced to the delivery side, adjust-

ments being made until a satisfactory reading on the oxygen manometer was obtained.

The water was allowed to circulate, by-passing the plate, for 15 minutes to allow the oxygen concentration to build up. The sample flasks were then placed in position, the blower started, and the water introduced to the plate. The air and water flows were adjusted to the required values. Care was taken to eliminate immediately all air from the sample flasks. Failure to do this led to the formation of minute air bubbles which were afterwards difficult to eliminate.

When equilibrium had been reached after a period of two hours (determined in a manner similar to that described previously), the instrument readings were recorded, together with the barometric pressure.

Preliminary runs were undertaken to ascertain a satisfactory flow of oxygen. This was done by trial and error. Care was taken to ensure that the inlet concentration of oxygen was safely below saturation so that no free oxygen entered the plate.

Materials and Analysis

The purity of solvents used in the investigation was as follows:

Methyl*cyclo*hexane.	Refractive index	20° C at 1·4231
Toluene . .	Refractive index	20° C at 1·4966
Methanol . .	Density	20° C at 0·7950 g/ml

The methods of analysis and sources of data for the different systems were:

Methyl*cyclo*hexane–toluene	Analysis by refractive index at 20° C in a Pulfrich refractometer. Data of Hall [8] for refractive indices and vapour–liquid equilibria.
Methanol–water	Analysis by refractive index at 20° C in a Pulfrich refractometer. Data presented by Perry [9] for density and vapour–liquid equilibria.
Oxygen–water	Analysis by modification of Winkler method.[10, 11] Data of White [12] for equilibrium data on oxygen solubility in water.

Condensed steam was used and found to be satisfactory for the oxygen desorption tests.

DISTILLATION COLUMN EXPERIMENTAL RESULTS

The superficial vapour velocity varied from 2 to 7 ft/sec for methyl*cyclo*hexane–toluene and from 1·5 to 3·0 ft/sec for methanol–water. The efficiency results for these two systems were, in the main, computed as overall efficiencies using the McCabe–Thiele [13] graphical method. However, as the work was carried out under total reflux, and the range of concentration covered was such that the average slope of the equilibrium curve m approached unity, the overall efficiency can be considered approximately equal to the average Murphree plate efficiency.[9]

For several runs in the methyl*cyclo*hexane–toluene system individual plate efficiencies were obtained and compared with the overall efficiencies with an average error of 2·0 per cent.

The efficiency data are tabulated in Tables I and II and plotted in Fig 3.

TABLE I

Overall Efficiency

(Methyl*cyclo*hexane–Toluene)

Run No.	Boil-up rate		Overall efficiency	
	G, lb/hr/ft²	V, ft/sec	Liquid samples, %	Vapour samples, %
1	1525	2·2	53·4	55·8
2	1780	2·6	55·8	56·8
3	2120	3·1	57·0	54·0
4	2250	3·3	66·0	69·0
5	2550	3·7	69·0	71·4
6	2975	4·3	75·7	77·0
7	3055	4·4	72·0	73·0
8	3100	4·5	75·2	77·5
9	3190	4·7	80·0	81·6
10	3250	4·7	79·0	81·2
11	3530	5·2	78·4	80·6
12	3635	5·3	80·0	81·2
13	4040	5·9	74·6	83·4
14	4120	6·0	74·8	74·0
15	4370	6·2	73·7	74·0
16	4580	6·7	74·4	73·0
17	4670	6·9	74·0	72·0

TABLE II

Overall Efficiency

(Methanol–Water)

Run No.	Boil-up rate		Overall efficiency
	G, lb/hr/ft²	V, ft/sec	Liquid sample, %
18	276	1·4	32·0
22	422	1·9	45·2
25	530	2·6	49·0
46	582	2·9	54·0

For methyl*cyclo*hexane–toluene the efficiency increases up to 5 ft/sec with a maximum of 80 per cent, while with a further increase in vapour velocity the efficiency decreases. The efficiency is sensitive to the boil-up rate, although it is relatively constant over the vapour velocity range of 4·3–6 ft/sec. The decrease in efficiency at the higher boil-up rates is thought to be due to entrainment. In the single-plate unit the amount of entrainment removed by the cyclone separator was measured, and the amount was such as largely to account for the decrease in efficiency at the higher vapour rates.

The reason for the limited number of results for the system methyl alcohol–water was due to the formation of rust in the column.

Liquid Film Efficiency Results for Single Tray Unit

The determination of liquid film efficiencies by the desorption of oxygen from oxygen-enriched water by

air was carried out over a range of superficial air velocities of 1·2–11·2 ft/sec and water rates of 0–6000 lb/hr/ft².

inlet and outlet temperature, together with a resultant small equilibrium solubility change across the tray, was inevitable.

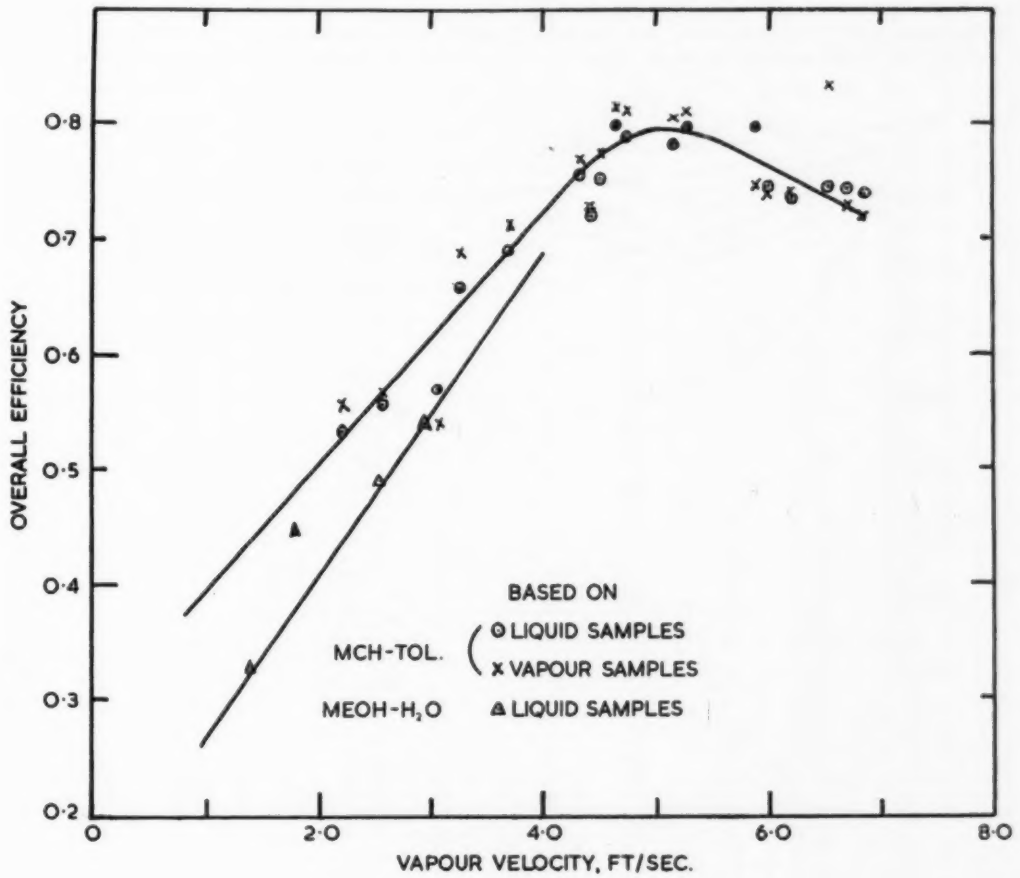

FIG 3

OVERALL EFFICIENCY *v.* VAPOUR VELOCITY

The liquid film efficiencies were computed from the relationship

$$E_L = \frac{X_{n+1} - X_n}{X_{n+1} - X_n^*} \quad . \quad . \quad . \quad (1)$$

where X_{n+1} = concentration of oxygen in p.p.m. entering;

X_n = concentration of oxygen in p.p.m. leaving the tray;

x_n = equilibrium concentration of oxygen in p.p.m. leaving the tray.

The values of x_n^* were based on the mean plate temperature. As no provision was made to hold the water on the test plate at the wet bulb temperature of the air, a slight temperature difference between the

Usually there was only a small difference in efficiencies based on mean and outlet temperatures, and it was only significant at low water and high air rates.

Over the complete series of runs the mean plate temperature, due to differing laboratory conditions, varied between 13° and 25° C. The correlation of Sherwood and Holloway [14] was used to correct the effect of temperature on the liquid film transfer coefficients.

The liquid film efficiency results are tabulated in Table III, and the corrected liquid film efficiencies plotted as a function of air and water rates are shown in Fig 4. From this figure it is seen that an increase of air velocity causes an increase in efficiency, while at low liquid flow rates an increase of water flow causes

a sharp decrease in the efficiency. The rate of decrease diminishes at higher liquid rates until the effect of further increase in water flow is negligible.

An increase in air velocity causes an increase of spray on the plate and, although differing radically from the mechanism of bubble plates, this results in an increase in aeration. This increase in aeration causes an increase in interfacial contact area, with consequent increase in efficiency.

At low air velocities (1·2 ft/sec) abnormally low liquid film efficiencies are obtained. This is due to unsatisfactory operation of the plate, as only one of the troughs is working correctly. The scatter of results for this air velocity is only to be expected, as the second trough bubbles spasmodically. This phenomena of one trough operating has been mentioned previously.[7]

A change in liquid rate will affect the performance of the tray, depending on the effect of two factors. These factors are the time of contact and the interfacial area of contact. Other things being constant, the time of contact will decrease with increased liquid flow. However, from visual observation, it was evident that considerably more spray was formed at the higher liquid rates than the lower ones. Thus, at higher liquid rates the latter factor tends to balance the former, with the result that the efficiency does not continue to decrease. The decrease in efficiency with increase in liquid rate is greater for the lower gas velocities.

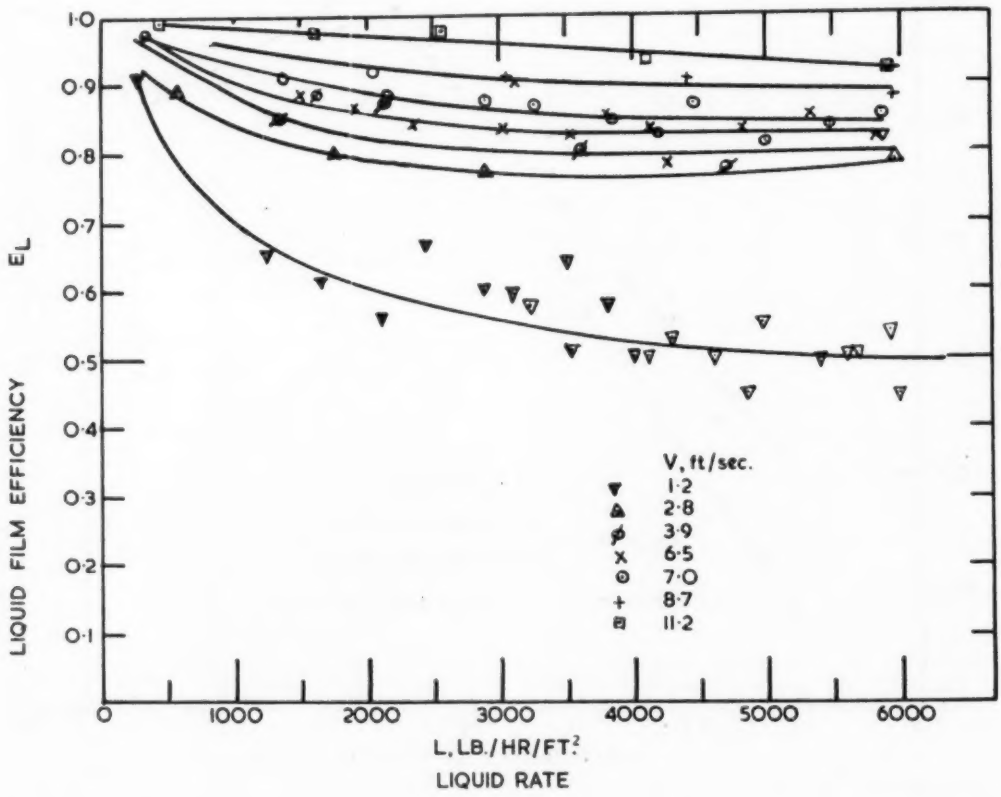

FIG 4

LIQUID EFFICIENCY *v.* LIQUID RATE

Liquid film transfer units, shown on Table III, were calculated from the Murphree liquid film efficiencies by equation (2)

$$-2{\cdot}3 \log (1 - E_L) = N_{Lt} \quad . \quad . \quad (2)$$

The data as presented by Perry[9] were used to construct a graph of $\frac{N_L\ 20^\circ\ \mathrm{C}}{N_{Lt}}$ *v.* temperature which was used to determine the N_L 20° C results given in Table III. The number of transfer units at 20° C is plotted as a function of liquid and gas rates on Fig 5.

As in the case of the efficiency results, the number

TABLE III

Liquid Film Efficiencies

(Desorption of Oxygen)

Run No.	Air velocity, ft/sec	Liquid rate, lb/hr/ft²	Mean plate temp, t° C	Oxygen concentration, p.p.m.			Atmos. press., mm Hg	Efficiency		Transfer units	
				x_{N+1} IN	x_n OUT	x_n^*		E_{L_t}	$E_{L_{OG}}$	N_{L_t}	$N_{L_{OG}}$
1	1·2	288	20·5	20·00	9·60	8·67	744	0·916	0·914	2·47	2·45
2	1·2	1240	20·3	25·80	14·50	8·65	741	0·657	0·654	1·07	1·06
3	1·2	1630	23·9	22·75	19·30	8·14	744	0·647	0·616	1·04	0·96
4	1·2	2070	23·8	25·80	15·40	8·20	746	0·592	0·563	0·90	0·83
5	1·2	2420	23·3	23·75	13·10	8·25	743	0·687	0·663	1·16	1·08
6	1·2	2870	21·7	22·25	13·85	8·60	753	0·616	0·602	0·96	0·92
7	1·2	3090	20·4	20·65	13·55	8·80	754	0·600	0·598	0·92	0·91
8	1·2	3250	24·0	20·35	12·85	8·05	738	0·610	0·578	0·94	0·86
9	1·2	3480	22·6	20·20	12·35	4·48	754	0·670	0·642	1·11	1·05
10	1·2	3520	20·9	25·40	16·65	8·70	749	0·524	0·517	0·74	0·73
11	1·2	3800	24·8	18·50	12·10	7·98	739	0·621	0·584	0·97	0·88
12	1·2	3970	22·9	23·40	15·45	8·25	736	0·525	0·505	0·74	0·70
13	1·2	4090	19·5	27·40	18·05	8·72	737	0·500	0·503	0·69	0·70
14	1·2	4250	22·2	25·80	16·75	8·38	741	0·550	0·532	0·80	0·76
15	1·2	4600	20·5	26·45	17·50	8·73	749	0·505	0·502	0·70	0·69
16	1·2	4810	19·4	22·25	16·05	8·40	706	0·448	0·450	0·59	0·60
17	1·2	4970	21·45	19·85	13·10	8·06	706	0·565	0·555	0·83	0·81
18	1·2	5380	21·8	27·00	17·60	8·64	758	0·513	0·500	0·72	0·69
19	1·2	5600	22·9	22·10	14·70	8·16	728	0·530	0·509	0·75	0·75
20	1·2	5660	21·6	23·90	15·80	8·38	734	0·522	0·511	0·74	0·72
21	1·2	5910	23·9	22·90	14·65	8·16	747	0·572	0·542	0·84	0·78
22	1·2	5950	21·8	25·40	18·35	8·48	745	0·440	0·428	0·579	0·56
23	2·8	575	17·85	24·35	11·30	9·40	762	0·881	0·892	2·13	2·22
24	2·8	1750	18·4	23·05	12·00	9·04	743	0·788	0·800	1·55	1·60
25	2·8	2900	21·6	22·35	11·50	8·50	744	0·783	0·773	1·525	1·48
26	2·8	5940	18·8	20·55	11·40	8·82	733	0·780	0·788	1·51	1·55
27	3·9	1350	19·7	22·20	10·70	8·64	731	0·850	0·852	1·89	1·91
28	3·9	1620	19·0	25·30	10·80	8·92	744	0·884	0·889	2·15	2·19
29	3·9	2150	17·95	23·40	11·70	9·07	740	0·876	0·880	2·044	2·12
30	3·9	3610	19·9	20·55	11·00	8·70	740	0·805	0·805	1·63	1·63
31	3·9	4700	19·3	20·90	11·60	8·82	740	0·770	0·773	1·47	1·48
32	3·9	5860	19·1	23·00	11·10	8·86	741	0·827	0·832	1·75	1·78
33	6·5	1500	21·0	25·40	10·40	8·68	749	0·895	0·887	2·25	2·18
34	6·5	1910	16·05	26·45	12·10	9·53	745	0·848	0·869	1·88	2·03
35	6·5	2350	18·2	24·30	11·60	9·06	742	0·834	0·841	1·79	1·84
36	6·5	3020	13·4	28·15	13·80	10·10	745	0·795	0·838	1·58	1·82
37	6·5	3110	21·05	20·75	9·70	8·57	740	0·908	0·901	2·38	2·31
38	6·5	3540	15·00	28·30	13·40	9·73	746	0·801	0·834	1·61	1·79
39	6·5	3800	18·95	23·45	10·95	8·84	738	0·855	0·861	1·93	1·97
40	6·5	4120	16·25	25·30	12·50	9·60	754	0·816	0·838	1·69	1·82
41	6·5	4250	13·25	26·25	14·50	10·20	747	0·732	0·783	1·31	1·52
42	6·5	4810	16·65	25·25	12·35	9·43	748	0·815	0·833	1·68	1·79
43	6·5	5320	18·05	24·40	11·55	9·18	748	0·843	0·854	1·85	1·92
44	6·5	5830	18·85	23·90	11·75	9·03	750	0·816	0·821	1·69	1·72
45	7·0	336	19·5	20·95	9·35	9·08	762	0·978	0·979	3·81	3·85
46	7·0	1350	21·1	17·10	9·15	8·43	729	0·917	0·913	2·49	2·44
47	7·0	2040	19·3	19·20	9·60	8·74	734	0·918	0·922	2·50	2·55
48	7·0	2160	20·4	21·40	10·20	8·82	754	0·890	0·888	2·20	2·19
49	7·0	3270	17·75	19·40	10·60	9·05	734	0·850	0·864	1·90	1·99
50	7·0	2880	20·0	19·95	9·90	8·48	722	0·877	0·877	2·10	2·10
51	7·0	3840	18·8	26·10	11·90	9·14	756	0·840	0·847	1·83	1·88
52	7·0	4200	19·7	26·75	11·95	8·86	748	0·827	0·828	1·75	1·76
53	7·0	4460	21·1	24·50	10·70	8·78	750	0·880	0·871	2·10	2·05
54	7·0	5000	19·0	25·60	12·20	9·02	751	0·807	0·813	1·64	1·68
55	7·0	5470	20·15	24·20	11·30	8·80	750	0·837	0·837	1·81	1·81
56	7·0	5860	18·75	20·65	10·60	8·78	729	0·845	0·852	1·86	1·91
57	8·7	3040	15·6	26·10	11·40	9·44	733	0·887	0·910	2·18	2·40
58	8·7	4410	17·35	23·35	10·55	9·10	731	0·898	0·911	2·28	2·42
59	8·7	5940	17·95	24·80	11·00	8·96	731	0·870	0·880	2·04	2·12
60	11·2	432	16·5	21·65	9·60	9·58	758	0·997	0·998	5·80	6·23
61	11·2	1600	19·8	24·35	8·90	8·56	721	0·980	0·980	3·90	3·90
62	11·2	2540	18·7	25·70	9·05	8·70	721	0·980	0·982	3·90	4·00
63	11·2	4100	18·45	23·85	9·75	8·77	724	0·935	0·940	2·72	2·80
64	11·2	5910	19·9	21·95	9·55	8·54	726	0·922	0·922	2·55	2·55

of transfer units increases with air velocity and decreases rapidly with liquid rates to a value of approximately 2000 lb/ft², and is then substantially constant.

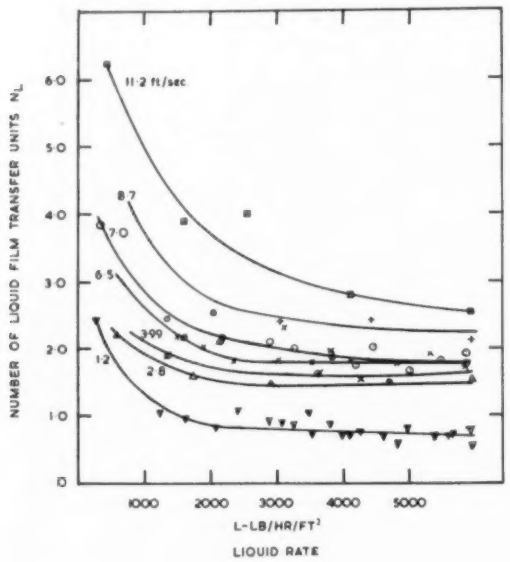

FIG 5
N_L v. L
(O_2 DESORPTION)

Calculated Gas Film Efficiencies

From the previously determined values of N_L and the overall vapour efficiency, the gas film transfer units N_G can be predicted for the systems methyl*cyclo*hexane–toluene, methyl alcohol–water, and ethyl alcohol–water,[7] using equation (3).

$$\frac{1}{2{\cdot}3 \log (1 - E_{oG})} = \frac{1}{N_G} + \frac{m\,G/L}{N_L} \quad . \quad (3)$$

where E_{oG} = overall gas or vapour efficiency;
N_G = number of gas or vapour film transfer units;
N_L = number of liquid film transfer units;
m = dy/dx or slope of equilibrium curve;
G = mass vapour velocity, lb/hr/ft²;
L = liquid rate, lb/hr/ft².

Equation (3) is for point efficiencies, and thus an approximation is made in using the experimentally-determined overall efficiencies. In the small tray used in this investigation there should be no great error involved in this assumption.

The number of liquid film transfer units for the system under consideration was calculated from the number of liquid film transfer units from the desorption of oxygen by the equation of Gerster.[15]

$$N_{L(1)} = N_{L(2)} \frac{D_{L(1)}^{\frac{1}{2}}}{D_{L(2)}} \quad . \quad . \quad (4)$$

Liquid film diffusivities for the binary systems were calculated using the method of Wilke.[16]

The calculated values of N_G and N_L for the three binary systems are plotted on Fig 6 as a function of superficial vapour velocity. For methyl*cyclo*hexane–toluene and methanol–water the $m\,G/L$ ratio was taken as unity, whereas for ethanol–water the value was 0·6.

Fig 6 compares the resistances due to the liquid and gas films. It will be noted that in all three cases the variation of N_L is small compared with that of N_G, and that N_G increases with increased vapour velocity or boil-up rate.

Consider first the curves for methyl*cyclo*hexane–toluene. At the low vapour velocities the gas film resistance, which is comparable to $\frac{1}{N_G}$, is greater than that of the liquid film. The liquid film resistance, however, is not insignificant.

As the boil-up rate is increased, the resistance of both films decreases, but that of the gas film decreases much more rapidly. When a velocity of 3·3 ft/sec is reached, the N.T.U. values and, as mG/L is unity, the resistance are equal.

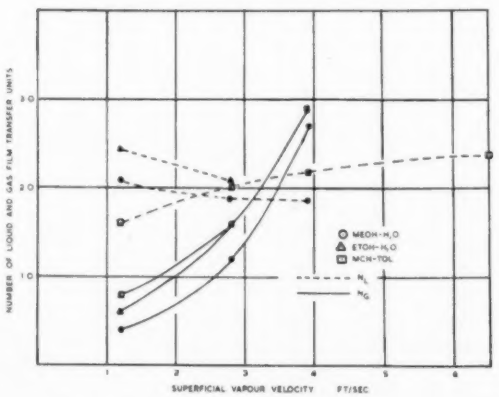

FIG 6

It is apparent, therefore, that the increase in efficiency with boil-up rate is due mainly to a decrease in the gas film resistance. Values of N_G have not been ascertained above the point where there is a reduction in efficiency due to entrainment.

The curves for the alcohol–water systems also show a decrease in the gas film resistances with increase in boil-up rate, but the liquid film resistances show small increases.

In making a comparison for the different systems at the same vapour velocity it is necessary also to consider the values of the liquid rate. A consideration of the vapour densities shows that for a given vapour velocity the volumetric liquid flow is considerably greater for methyl*cyclo*hexane–toluene than for ethanol–water and methanol–water. From Fig 5 it is seen that the reduction in the number of liquid transfer units with liquid rate is more evident at low liquid loadings and that this effect is greater than the increase in N_L due to increased vapour velocity. Thus for total reflux conditions :

(1) The gas film resistance is reduced with increased vapour velocity, giving the characteristic Kaskade plate increase of efficiency with boil-up rate.

(2) The liquid film resistance may increase or decrease with boil-up rate depending on the magnitude of the volumetric liquid flow.

CONCLUSIONS

Arising out of this investigation of two trough Kaskade tray units the following conclusions are reached :

(1) Overall and liquid film efficiencies increase with vapour velocity. In the case of methyl*cyclo*hexane–toluene a maximum efficiency of 80 per cent is obtained at a vapour velocity of 5 ft/sec. Above this velocity the efficiency decreases, and it is suggested that this decrease is due to entrainment.

(2) For this apparatus the overall efficiency is relatively constant over a vapour velocity range of 4·0–6·0 ft/sec.

(3) At constant vapour velocity the liquid film efficiency rapidly decreases with increasing liquid rate and then remains substantially constant.

(4) At total reflux the gas film is more controlling than the liquid film at low vapour velocities, but becomes progressively less controlling with increasing vapour velocity. The liquid film resistance remains relatively constant.

(5) At low vapour velocities low efficiencies are obtained, as one of the two troughs in the tray does not operate correctly.

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THE SEPARATION OF NAPHTHALENE AND ITS HYDROGENATED PRODUCTS BY VAPOUR-PHASE CHROMATOGRAPHY *

By R. M. SOEMANTRI † and H. I. WATERMAN † (Hon. Fellow)

SUMMARY

The analysis of naphthalene and its hydrogenated products by means of gas–liquid partition chromatography is described. A description of the apparatus is given. The choice of conditions and the influence of these on the separation are discussed. Solid mixtures can be liquefied before the analysis by heating or, more elegantly, can be " liquefied " by adding benzene to them, without disturbing the quantitative analysis.

The method of calibration and the quantitative interpretation of the chromatograms are discussed. The accuracy of the method is $\pm 1{\cdot}5$ per cent absolute. The chromatographic columns used prove to be stable, and no decline in separating efficiency can be observed. No dehydrogenation of the relatively unstable decalin and tetralin can be detected during the analysis.

INTRODUCTION

VAPOUR-PHASE chromatography, first suggested by Martin and Synge,[1] has now become a very important tool for the analysis of volatile compounds. Applications of this analytical separation technique to gas and liquid mixtures have been described by Ray,[3] Bradford *et al*,[4] van der Craats,[5] James,[6] James and Phillips,[7] Keulemans and Kwantes,[8] Lichtenfels,[9] and several others.

Vapour-phase chromatographical methods can be divided into two essentially different groups :

(1) *Gas–solid adsorption chromatography*,[10] employing a solid adsorbing agent. This method can be sub-divided into two groups—

(*a*) Separation by *displacement* : the gaseous mixture is adsorbed on to active charcoal or silica gel and then desorbed by raising the temperature, or it is displaced by a vapour that is more strongly adsorbed.

(*b*) Separation by *elution* : the adsorbed components are eluted by means of an inert stripper gas,[3, 11] mostly N_2 or H_2.

(2) *Gas–liquid partition chromatography*, employing a liquid agent for partition. This paper deals only with this latter method.

PRINCIPLE

In gas–liquid partition chromatography the moving phase is a gas–vapour mixture, and the stationary phase consists of a non-volatile, high-boiling liquid, supported on an inert solid carrier. Due to the difference in partition coefficient of the several components, they will be separated and leave the column one after another by stripping with the carrier-gas.

METHOD OF DETECTING

Several detecting devices have been developed and are suitable for detecting and measuring the separated components :

(1) measuring of thermal conductivity;
(2) measuring the pressure increase after condensing or chemically binding the carrier gas;
(3) automatic titration (for acids); [2]
(4) β-ray ionization cell.

The measuring of thermal conductivity is relatively simple, sensitive, and reliable.

DESCRIPTION OF THE APPARATUS

In the present apparatus the thermal conductivity cell (" katharometer ") is of the Bureau of Mines type.[12] It consists principally of a cylindrical copper block, in which are two parallel bores of 3 mm diameter. In these two bores are stretched two identical Pt wires of 0·020 mm diameter, 10 cm in length. Both wires form two arms of a Wheatstone bridge and are fed with a 100 mA current from an accumulator. Through one of these bores (reference channel) streams the carrier gas only, while through the other (comparative channel) streams the carrier gas and the separated component. Due to the difference in thermal conductivity in both bores, the Wheatstone bridge will be out of equilibrium and the overpotential is fed to an automatic Brown potentiometer (0–2·5 mV), coupled with a pen recorder.

The carrier gas N_2 is drawn from a bomb via a reducing valve. As a constancy of the gas flow is essential for the chromatogram, N_2 is led to a precision reducing valve (Negretti and Zambra). An accurate and constant pressure drop can thus be maintained over the column. In order to eliminate any pressure variations, a buffer vessel is attached after the

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precision reducing valve. The column and the katharometer are immersed in the oil bath, so that they have the same temperature. The column is coiled into a spiral in order to save space.

The gas flow is measured by means of a rotameter. The inlet system consists of a valve, over which a rubber serum-cap is attached. In this way leakage of the system during the injection of a sample is eliminated. A schematic flow-sheet of the apparatus is given in Fig 1.

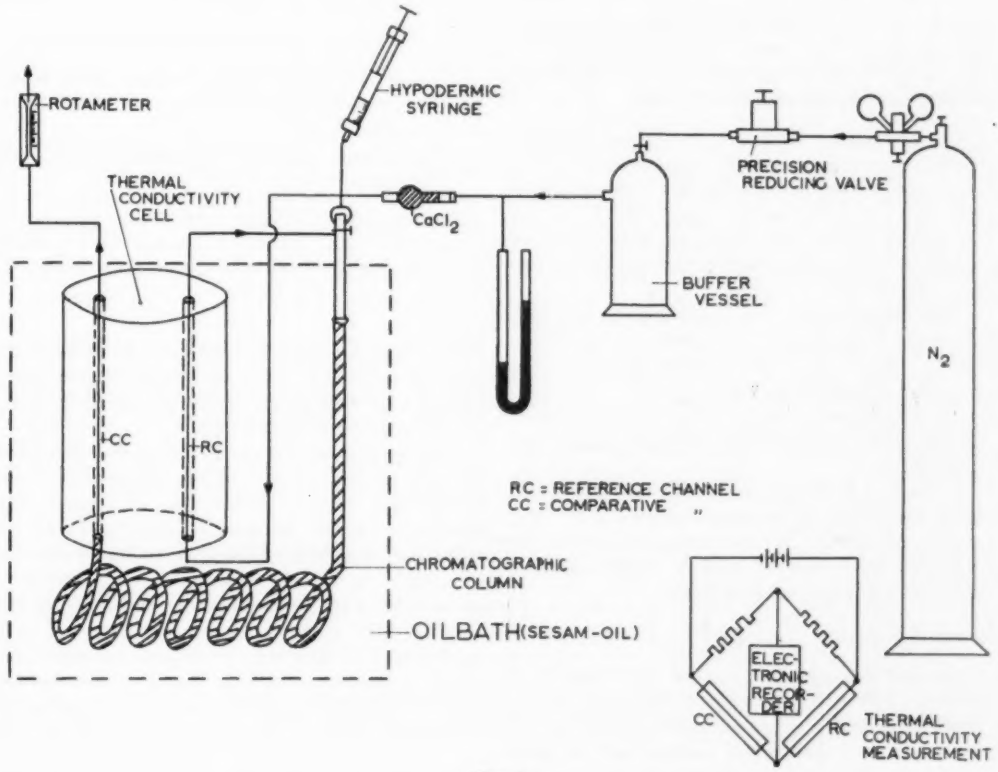

FIG 1

THE SEPARATION

Martin [2] and others have given a theory of gas–liquid partition chromatography. This theory gives indications as to how to achieve a good separation, but due to the fact that a large number of factors play a part in the process, it is still difficult to predict the optimum conditions for each problem.

Some of the important factors for separation are [5]:

(1) The nature of the absorption liquid and the column temperature, as these determine the Henry coefficient and therefore the partition coefficient;

(2) the velocity of the carrier gas, column diameter, size of the sample, and type of the solid carrier.

It is beyond the scope of this paper to deal comprehensively with these factors. For a fuller account one is referred to [4, 5, 8, 9].

EXPERIMENTAL PART

Separation of Naphthalene

The intention is to study the separation of naphthalene and its hydrogenated products for the analysis of the products obtained in semi-technical dehydrogenation processes of decalin.

Table of Components

	B. p., °C
Naphthalene	217·9
Decahydronaphthalene (decalin):	
trans	185·5
cis	194·6
Tetrahydronaphthalene (tetralin)	207·2

For this purpose several accurately-weighed mixtures of these components were prepared and analysed by means of gas–liquid partition chromatography. Before analysing these mixtures all the components were first tested for purity (see Table I).

Choice of Conditions of Analysis

(1) *The Chromatographic Column:* analyses were carried out on two kinds of column:

(*a*) Dow Corning Silicone 702, impregnated on Celite, column diameter 12 mm.

(*b*) dioctylphthalate, impregnated on ground firebrick, column diameter 6 mm.

The column length is 180 cm (coiled copper tube).

These columns have been used successfully for analyses of hydrocarbon gases and gasoline fractions.

The columns were prepared as follows:

(*a*) Kieselguhr (Celite 545, Johns Manville Corpn) was size-graded by repeated suspension in water, heated in a muffle for three hours at 300° C, and purified by treatment with HCl 1·19, to remove iron and basic impurities. After washing with water until acid-free, Celite was dried at 145° C.[2]

The liquid phase, consisting of Silicone 702, dissolved in pentane, was added to the Celite (0·5 g silicon/1 g Celite), stirred well, and the pentane evaporated at 70° C.

The mixture is packed down to a known density (0·4 g/cc) and the two ends plugged with glass-wool.

(*b*) Screen fractions of firebrick (40–100 mesh), after suspension in water to remove dust, and drying at 150° C, were impregnated with di-octylphthalate, diluted with a volatile solvent (0·3 g dioctylphthalate/1 g brick).

(2) *The Operational Variables:*

(*a*) The *temperature*: a few analyses were done at 140° C, the others at 170° and 180° C. The influence of the temperature on the shape of the peaks and the time of analysis is illustrated in Figs 2 and 3. The lower the temperature, the broader the peaks and the longer the time of analysis. At 170° C the peaks are relatively sharp and symmetrical and sufficiently separated for quantitative work.

Of course a higher temperature can be chosen for these analyses in order to shorten the time of analysis and to improve the efficiency of the column,[2] but this is limited to a certain extent:

(i) At higher temperatures the silicone or dioctylphthalate have still higher vapour pressures, so they can be stripped by the carrier gas. The column will be in this way seriously damaged.

At 170°–180° C no decline could be detected in the separating efficiency of our columns.

(ii) As tetralin and decalin are relatively unstable, very high temperatures have to be avoided in order to prevent any dehydrogenation of these components at the hot Pt wire or at the walls of the bores of the katharometer.

(*b*) *Velocity* of the carrier gas: 5·5 l/hr N_2 for the dioctylphthalate; 6·5 l/hr N_2 for the silicone column.

(*c*) *Size* of the liquid samples: 0·15–0·20 ml.

The limitations of the size of the samples are:

upper limit: the column must not be overloaded and the peaks must not run out of the recorder scale.

lower limit: determined by the sensitivity of the katharometer.

According to Ray,[3] the samples were put on to the chromatographic column by means of a calibrated tuberculin syringe provided with a long needle.

Note

The mixtures that were not liquid were liquefied by heating at 70° C. From the homogeneous liquid a sample was then taken in the preheated syringe, in order to prevent solidification.

This method has the disadvantage that there might be loss of the most volatile component, due to evaporation during the heating. The most elegant method is to add benzene or other solvent to the solid mixtures until a homogeneous liquid solution is obtained. In this way heating is not necessary, and so losses due to evaporation are avoided. It is not necessary to calculate the amount of solvent added, as it does not interfere or influence the ratio of the other components. Fig 5 shows a chromatogram obtained in these analyses and Fig 6 shows a chromatogram of a mixture to which benzene was added.

Qualitative and Quantitative Interpretation of the Chromatograms

Qualitative: the retention volume, or at a given velocity of the carrier gas, the retention time of a component, is determined by the nature of the stationary phase (absorption liquid), the temperature, and the velocity of the carrier gas. At constant operating conditions, the retention time (volume) of a component is specific and constant (R_v). A component can thus be identified by means of its retention time.

Quantitative: the area of a peak is directly proportional to the quantity of the corresponding component.[4, 5, 13] In most cases it is not necessary to measure the actual peak area, but it is sufficient to measure it by multiplying the height by the half-band width of the peak ($a \times b$).

If the thermal conductivities of the components are equal, or when the differences are negligible, then

the area of a peak is directly proportional to the mol-concentration of the corresponding component. According to Phillips,[13] this will be the case when hydrogen or helium are used as carrier gas. These have very high thermal conductivity in comparison with other vapours and gases.

The total sum of the areas of the peaks is taken as 100 per cent and the ratio between the peak area and this sum gives the mol-percentage of the corresponding component. In this case, helium being unavailable, N_2 is used as carrier gas. The thermal conductivity of N_2 differs little from that of the other vapours.

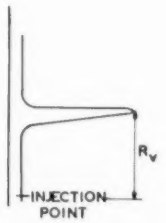

FIG 2 (*a*)
QUALITATIVE

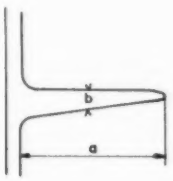

FIG 2 (*b*)
QUANTITATIVE

In following the method described above, the values found differ considerably from the values calculated from the preparation of the mixtures.

The values found for naphthalene were all too high and deviations as large as 6·6 per cent absolute were encountered. Thus it was necessary to carry out calibration and to make some corrections. Evidently, in using N_2 as carrier gas, the difference in thermal conductivity of the components is not negligible.

Methods of Calibration

(1) According to Bradford *et al*[4] by the " internal standard " method. This method has the following disadvantages: first, it is very difficult to add an accurate fixed amount of the " internal standard " to all mixtures to be analysed; secondly, several calibration mixtures must be prepared and analysed to obtain accurate calibration lines for all components.

(2) Another method is to measure the peak area corresponding to an accurately known amount of a component. This method is rather time-consuming and complicated; the peak area is influenced by the operation temperature, and it is not possible to inject an accurate amount with a common syringe.

(3) The method of calibration used in these analyses is as follows: equimolar mixtures of decalin and tetralin and equimolar mixtures of tetralin and naphthalene were prepared. Assuming equality of

TABLE I

Mixture	Mol, %											
	trans-Decalin			*cis*-Decalin			*Tetralin*			*Naphthalene*		
	c	*f*	*d*	*c*	*f*	*d*	*c*	*f*	*d*	*c*	*f*	*d*
Commercial decalin D_H	—	79·6	—	—	20·4	Very nearly the value found by analytical distillation and i.r. spectrography						
T_E = tetralin obtained by distillation	—	2·3	—	—	3·2	—	—	94·5	—	—	—	—
N = recrystallized naphthalene	—	—	—	—	—	—	—	—	—	—	100·0	—
T_H = commercial tetralin	—	—	—	—	—	—	—	100·0	—	—	—	—
cis-Decalin	—	—	—	—	100·0	—	—	—	—	—	—	—
Commercial decalin + *cis*-Decalin I	17·9	16·2	−1·7	82·1	83·8	+1·7	—	—	—	—	—	—
Commercial decalin + *cis*-Decalin II	46·5	47·9	+1·4	53·5	52·1	−1·4	—	—	—	—	—	—
D_HT_E II	39·2	39·0	−0·2	11·3	11·7	+0·4	49·5	49·3	−0·2	—	—	—
ND_H I	70·9	71·0	+0·1	18·1	18·9	+0·8	—	—	—	10·3	10·1	−0·2
NT_E I	1·9	2·5	+0·6	2·7	3·0	+0·3	83·4	80·5	−2·9	12·0	13·9	+1·9
NT_E II	1·0	1·3	+0·3	1·4	2·2	+0·8	46·3	47·0	+0·7	51·3	49·5	−1·8
D_HT_H I	36·2	34·4	−1·8	9·2	8·4	−0·8	54·5	57·2	+2·7	—	—	—
ND_HT_E I	46·9	44·5	−2·4	13·0	14·4	+1·4	38·4	38·1	−0·3	1·8	3·0	+1·2
ND_HT_E II	25·6	26·6	+1·0	7·3	8·3	+1·0	31·3	32·6	+1·3	35·8	32·5	−3·5
ND_HT_E III	7·2	7·0	−0·2	4·0	4·3	+0·3	85·1	83·5	−1·6	3·7	5·3	+1·8
ND_HT_E IV	3·2	2·0	−1·2	1·2	0·7	−0·5	14·8	14·3	−0·5	80·9	83·0	+2·1
ND_HT_E V	73·7	73·4	−0·3	19·0	19·2	+0·2	5·8	5·9	+0·1	1·6	1·5	−0·1
ND_HT_E VI	3·7	3·5	−0·2	2·2	2·6	+0·4	50·0	50·7	+0·7	44·2	42·2	−2·0
ND_HT_E VII	56·3	57·9	+1·6	14·5	17·0	+2·5	4·4	4·0	−0·4	24·8	21·2	−3·6
ND_HT_E VIII	31·3	31·4	+0·1	8·5	8·8	+0·3	20·4	21·3	+0·9	39·8	38·6	−1·2
ND_HT_E IX	32·7	32·5	−0·2	9·4	9·4	0	39·0	40·0	+1·0	18·9	18·2	−0·7
ND_HT_E X	13·8	15·0	+1·2	4·5	5·0	+0·5	36·9	38·0	+1·1	44·8	42·0	−2·8

c = calculated. f = found from chromatogram after correction. d = deviation.

thermal conductivities, the peaks of the components of such equimolar mixtures must have the same area.

Table of Calibration

	Prepared, %	Area of peak mm²	
Tetralin . . .	49·6	495·0	576·3
Naphthalene . .	50·4	660·0	756·0
Tetralin . . .	50·0	2065·5	3333·0
Decalin (*cis* + *trans*) .	50·0	1900·6	3171·8

From this table it is clear that naphthalene gives a larger area than tetralin, and decalin gives a smaller area than tetralin. Therefore the areas of the components are not directly proportional to their mol-concentrations, but the following corrections must be applied.

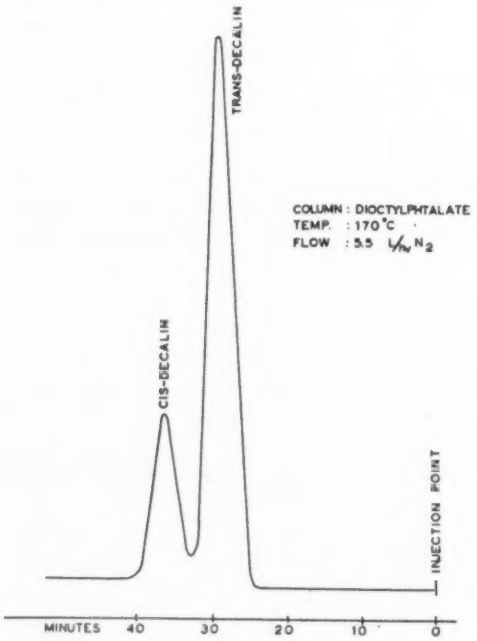

Fig 3

Table of Correction

Area of tetralin	× 1
Area of naphthalene	× 1/1·3
Area of decalin	× 1·07

By applying these correction factors in interpreting the chromatograms, it is found that the values (mol-percentages) obtained for the components are correct. The mean deviation is about 1·5 per cent absolute (see Table I). This method of calibration is relatively accurate and simple compared with other

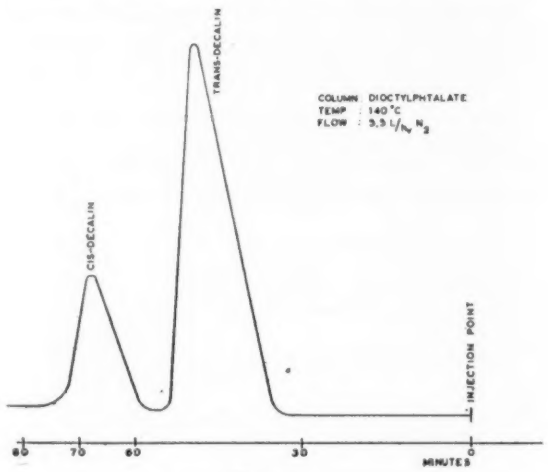

Fig 4

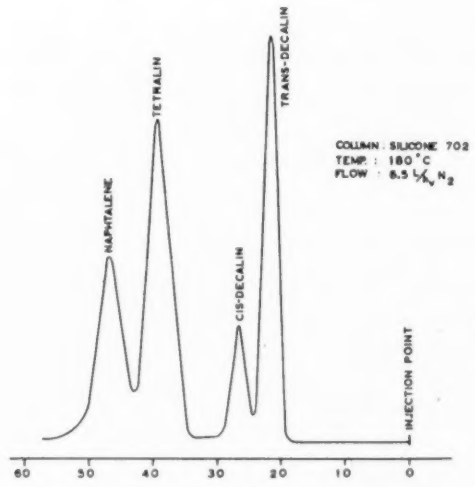

Fig 5

calibration methods. The ratio of the peak areas of the components is temperature-independent in the temperature range 140°–180° C.

CONCLUSIONS

(1) On both columns the components are readily separated and leave the column according to their boiling points.

(2) No influence of the column diameter on the separating efficiency can be observed. Apparently no disturbing radial diffusion occurs. Both columns are stable at these operating conditions.

(3) Gas–liquid partition chromatography offers a simple, rapid, and accurate method of analysis for these high-boiling, sometimes solid, mixtures, and

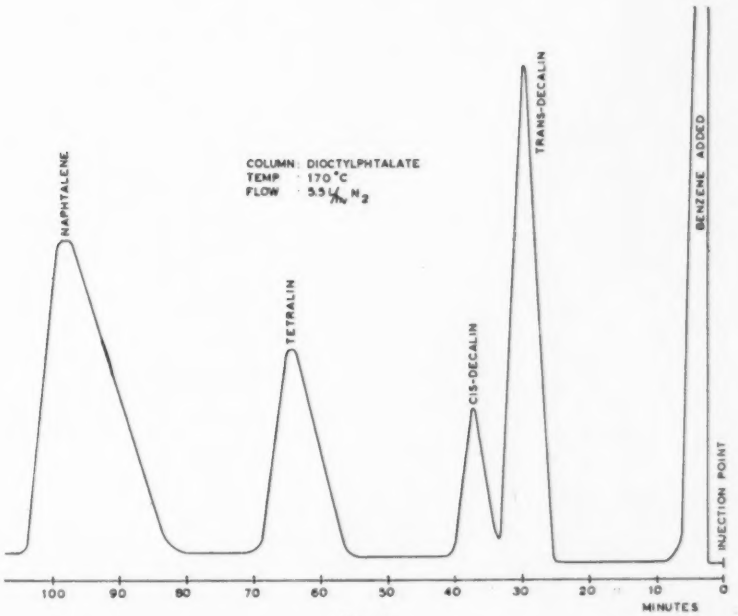

FIG 6

requires only very small samples (0·10–0·20 ml). This method has great advantages over other possible methods, like analytical distillation or combined ultra-violet and infra-red spectroscopy.

(4) No dehydrogenation of decalin and tetralin can be detected. Above 200° C some dehydrogenation might occur, which will render the analysis inaccurate and complicated.

(5) Concentrations as small as 1 per cent can be detected quantitatively.

ACKNOWLEDGMENTS

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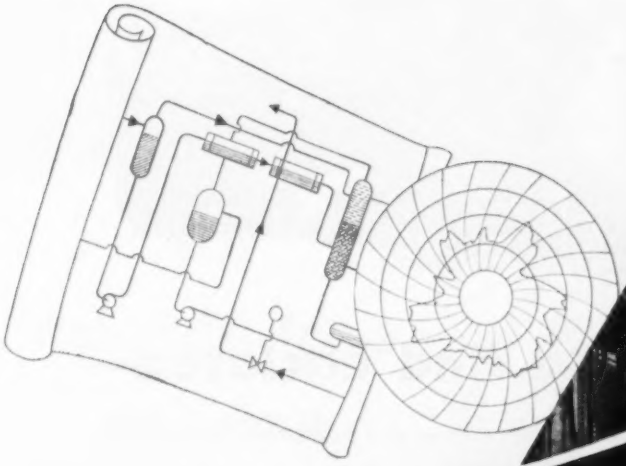

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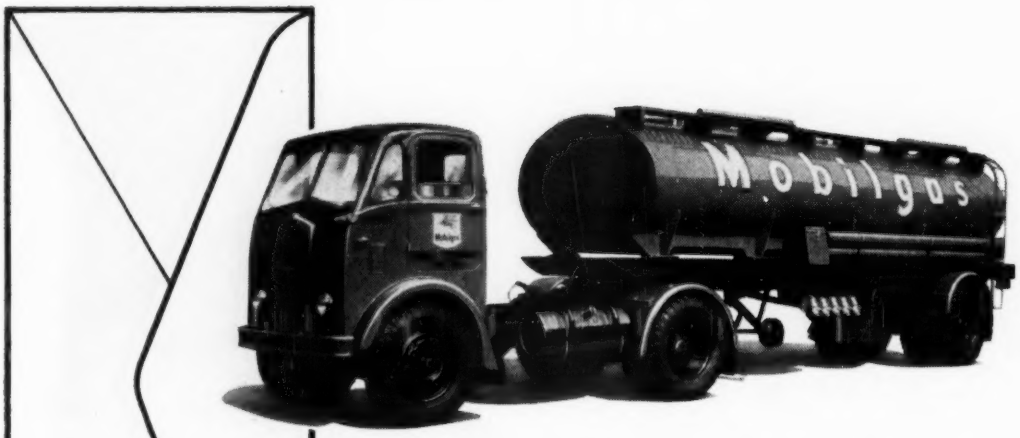

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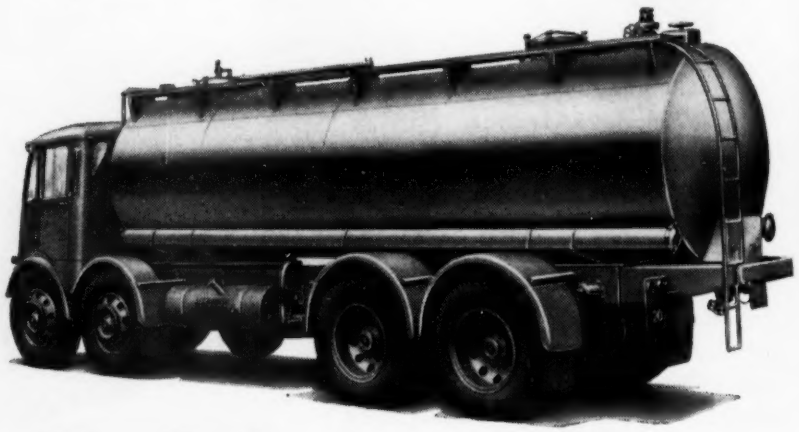

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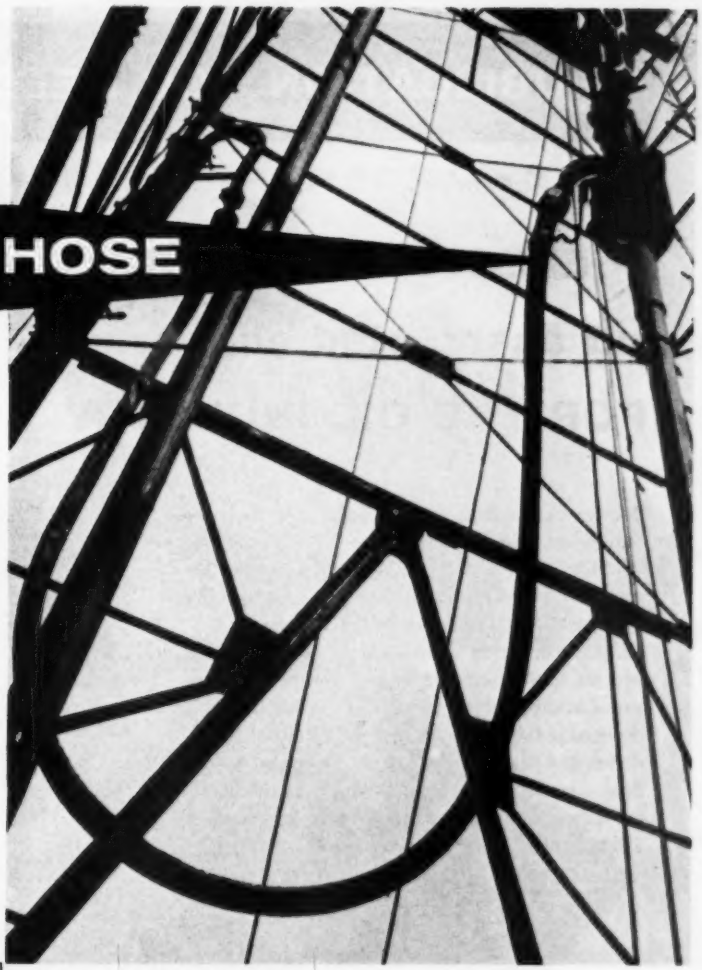

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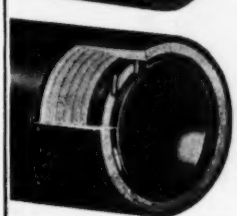

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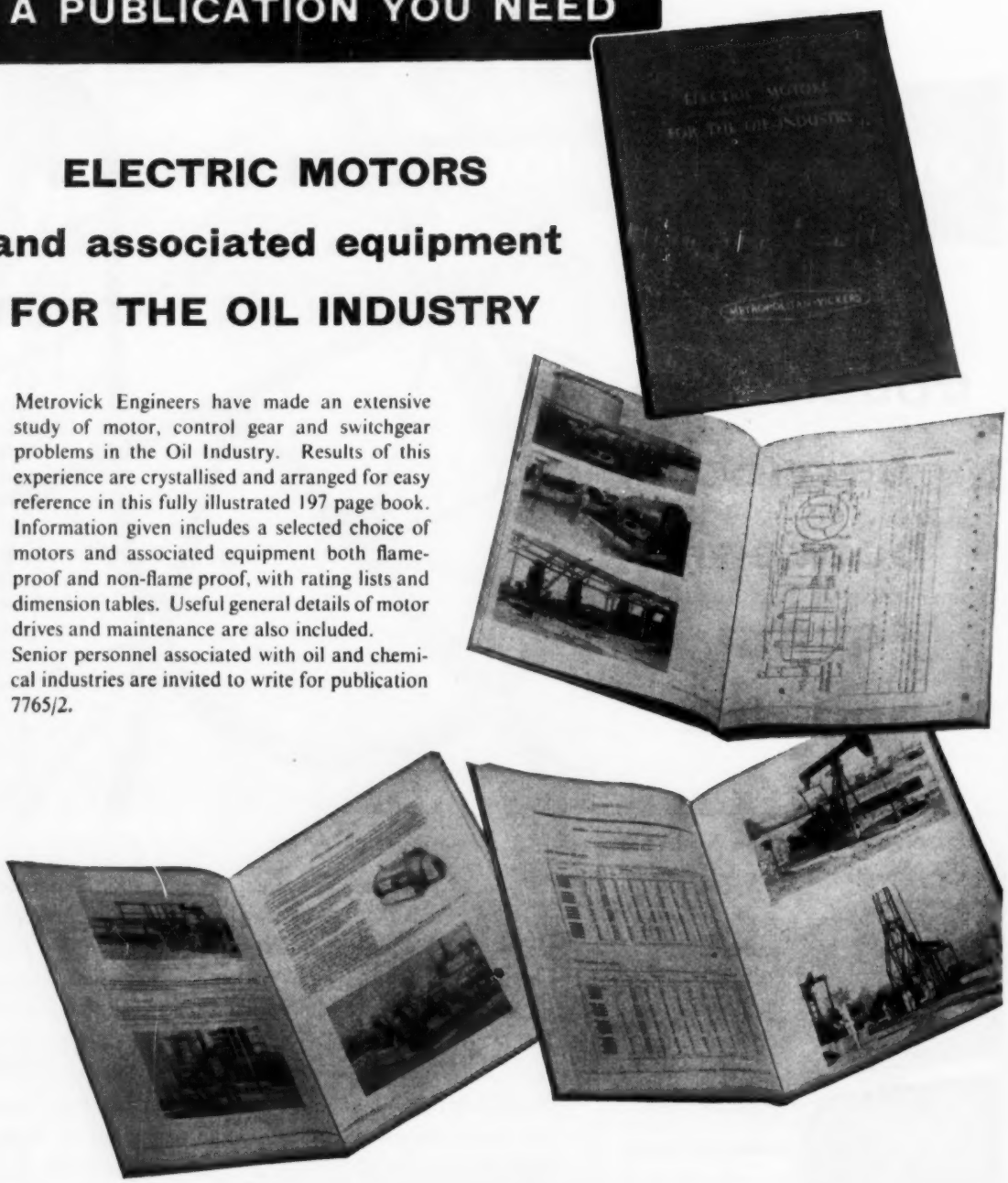

Metrovick Engineers have made an extensive study of motor, control gear and switchgear problems in the Oil Industry. Results of this experience are crystallised and arranged for easy reference in this fully illustrated 197 page book. Information given includes a selected choice of motors and associated equipment both flame-proof and non-flame proof, with rating lists and dimension tables. Useful general details of motor drives and maintenance are also included.
Senior personnel associated with oil and chemical industries are invited to write for publication 7765/2.

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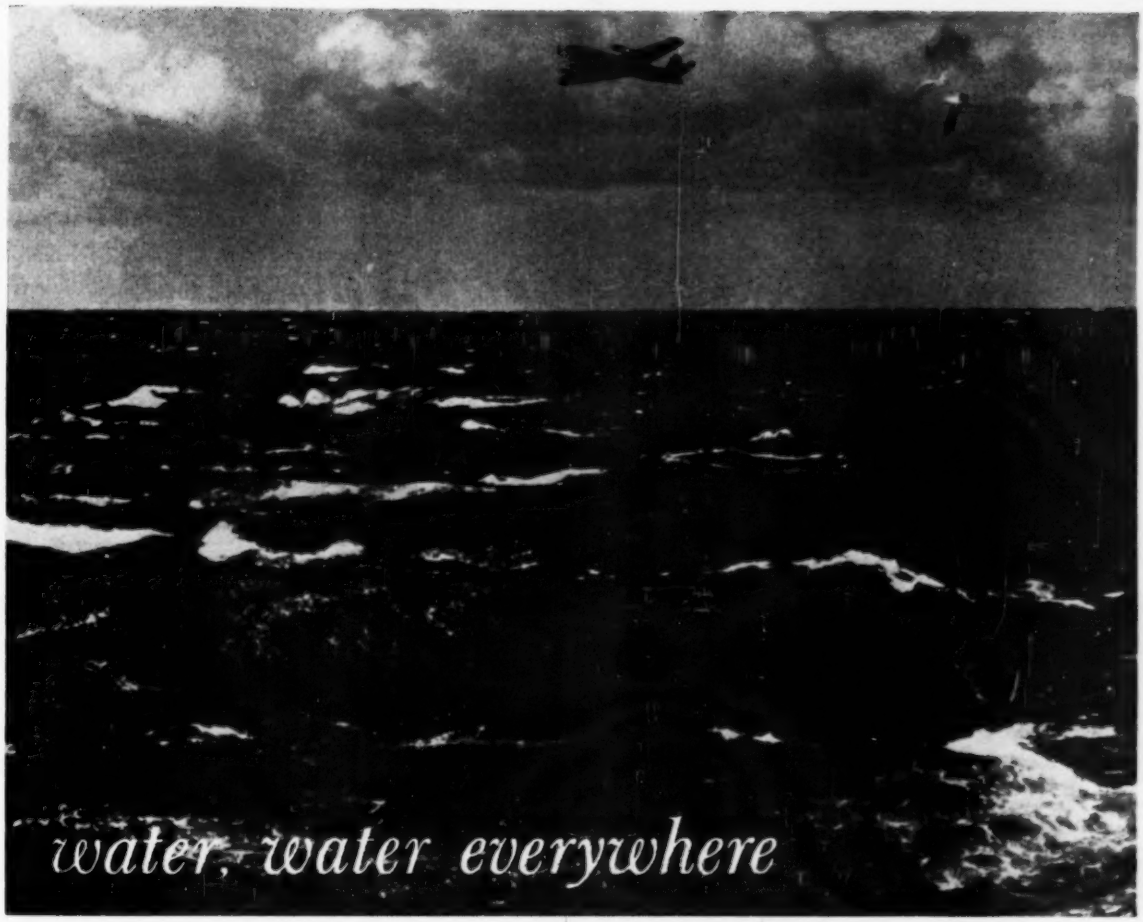

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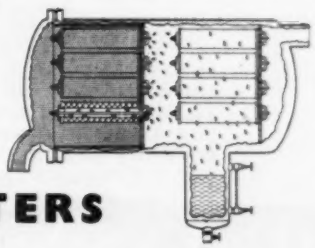

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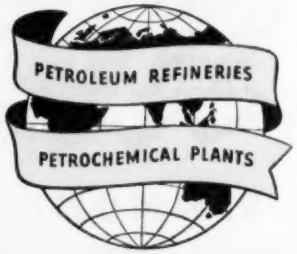

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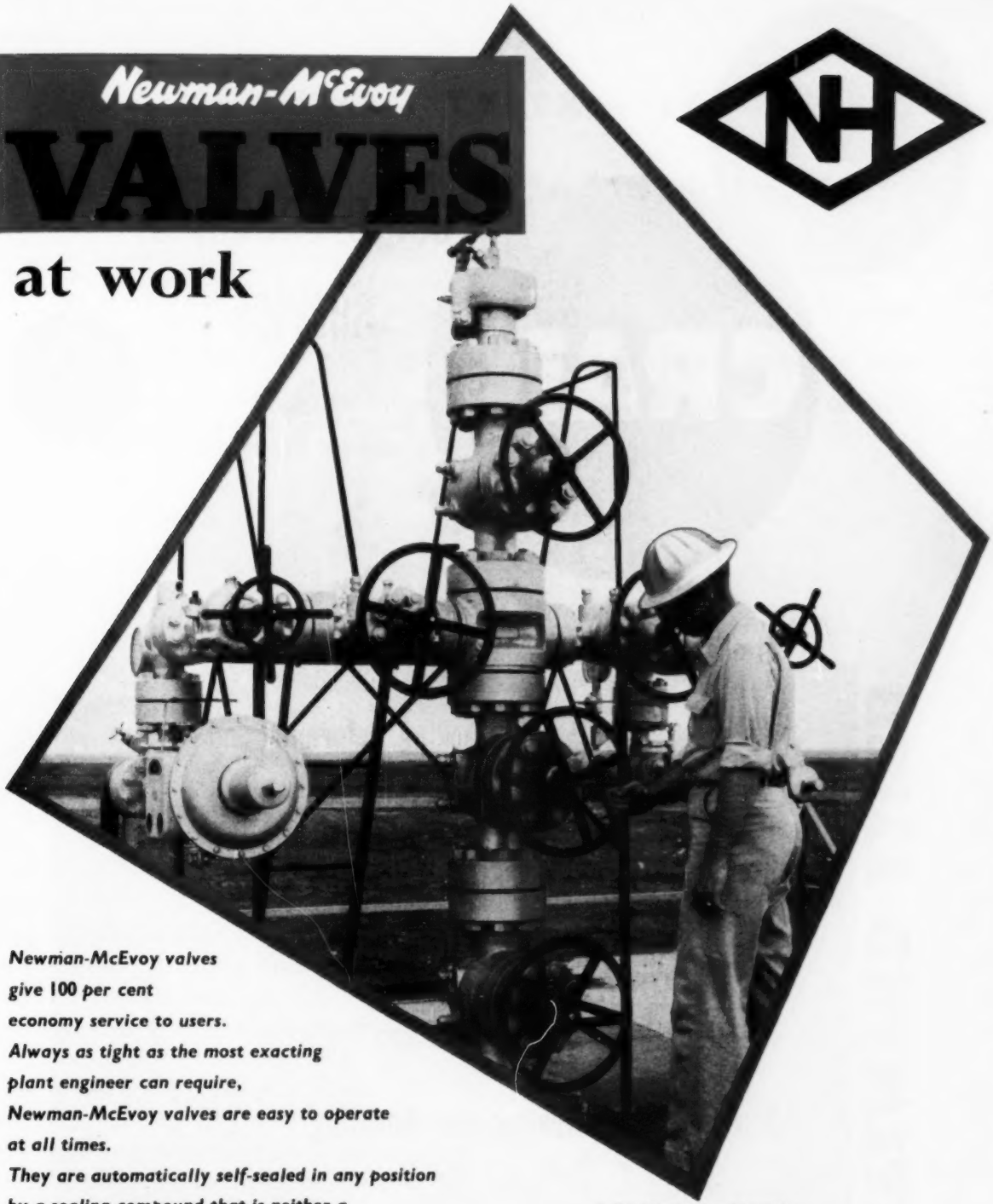

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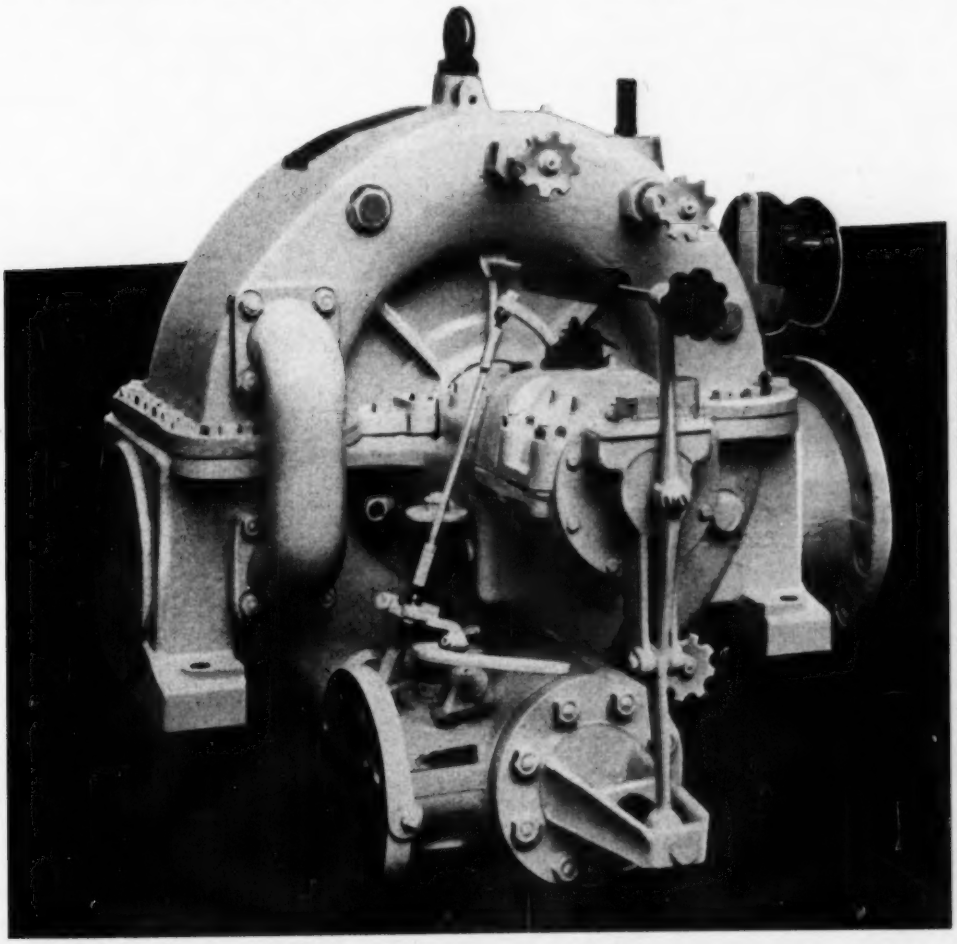

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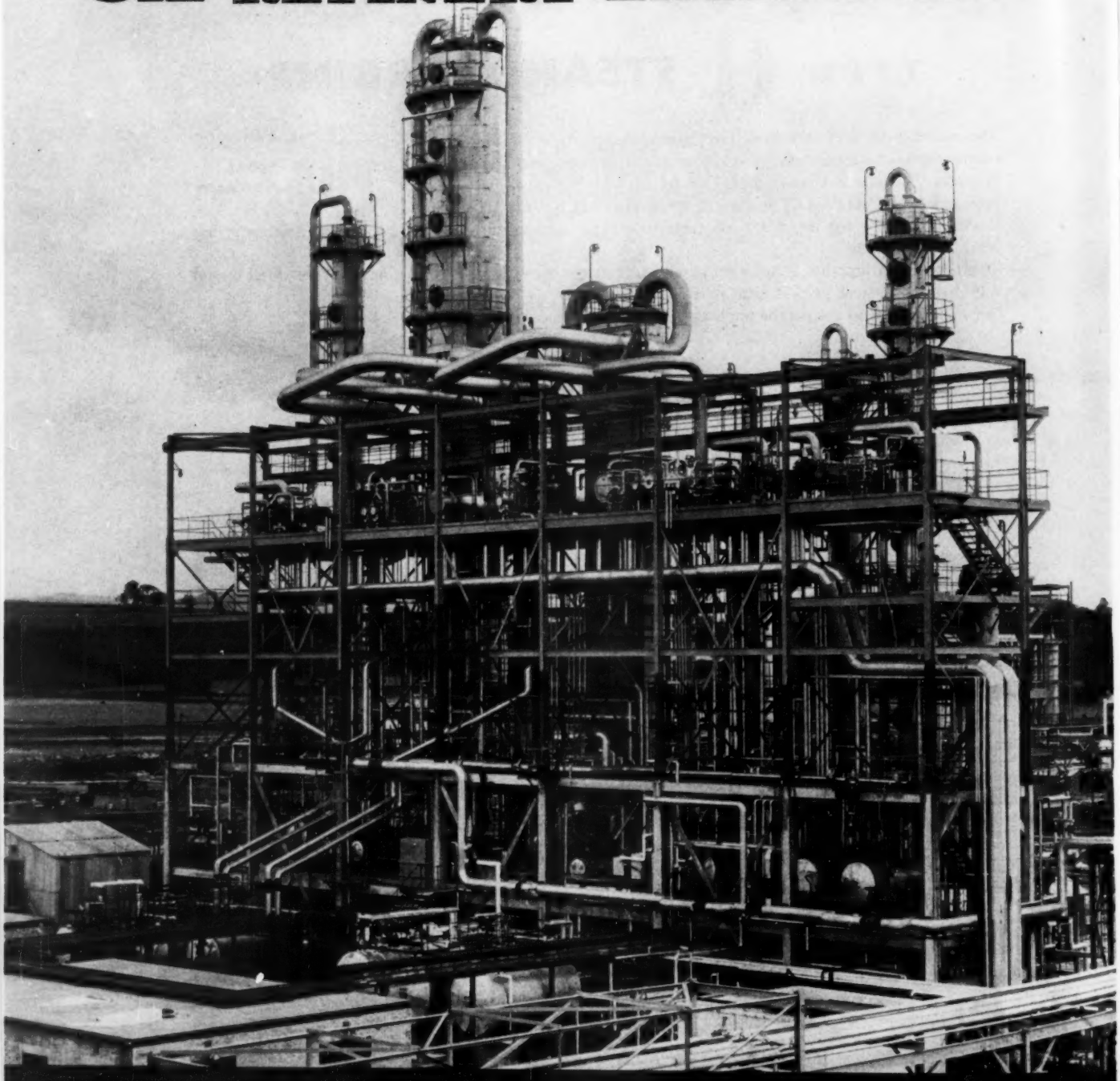

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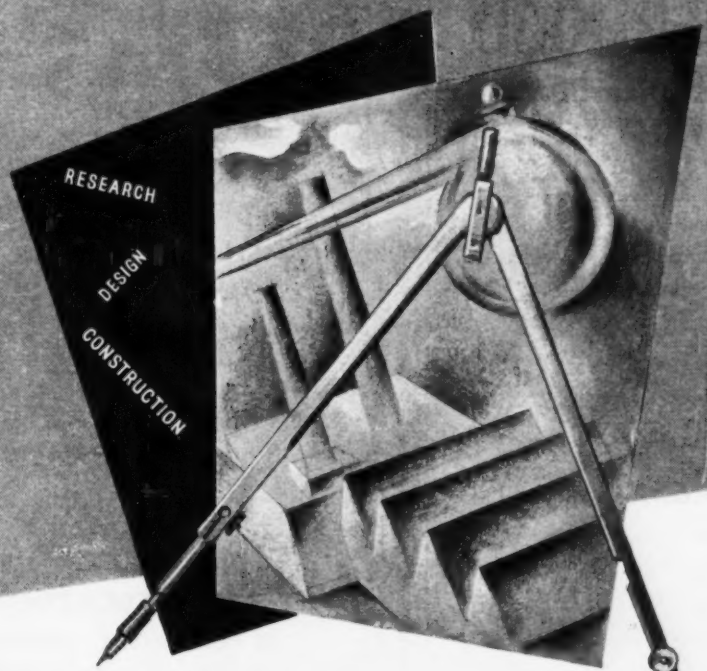

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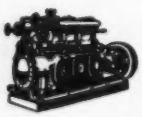

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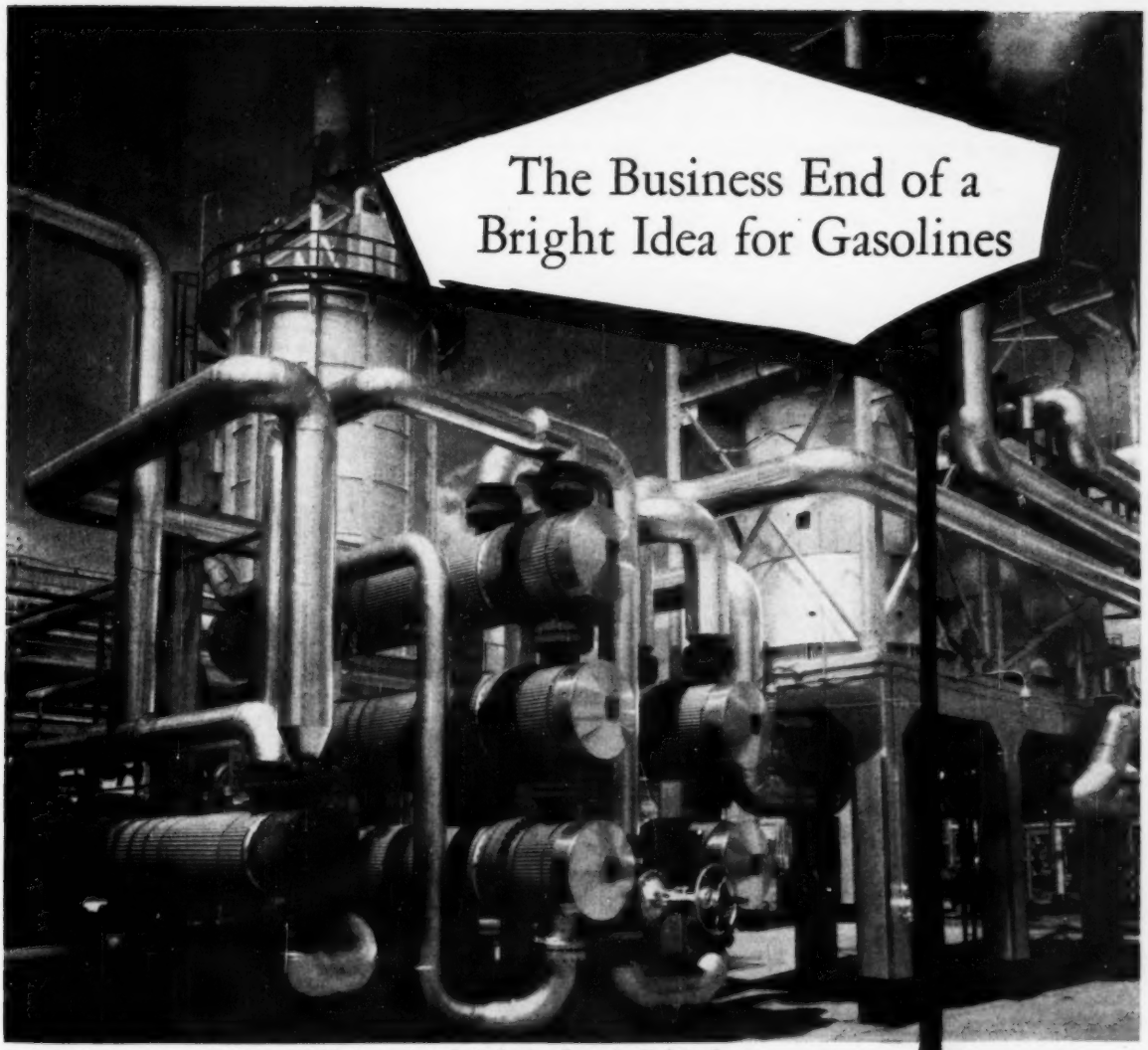

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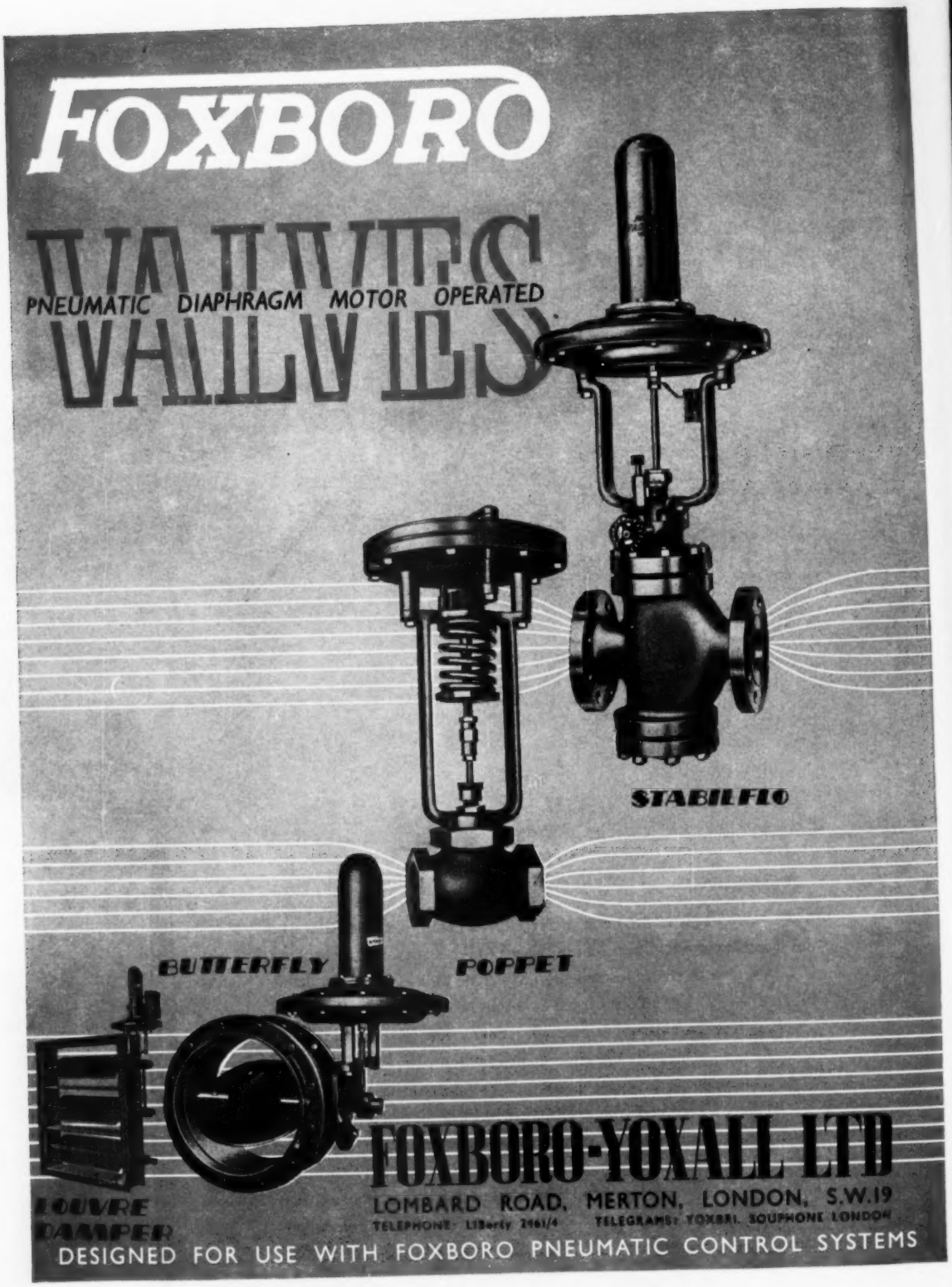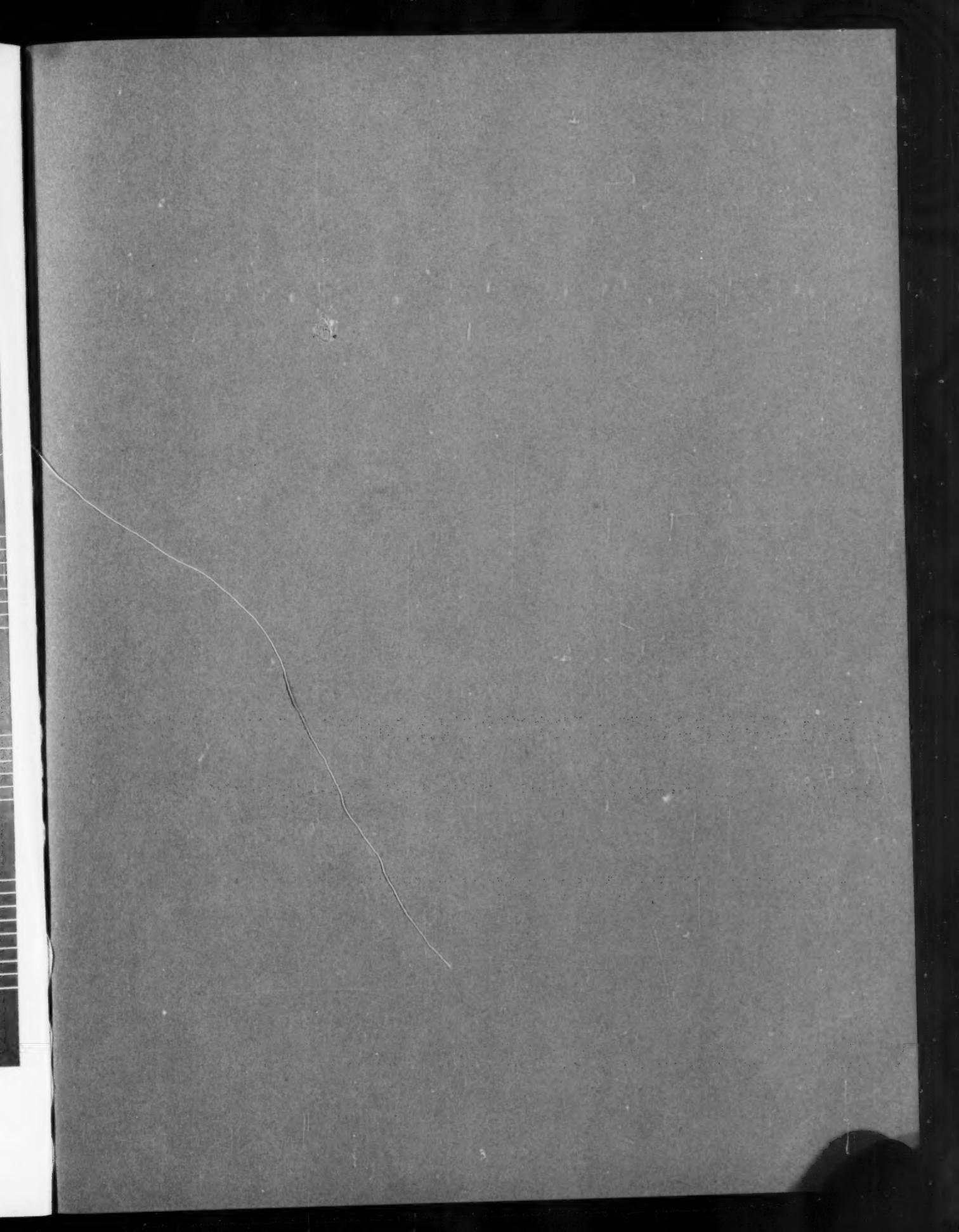
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